

FINAL REPORT
VOLUME II

P. J. ...

STATISTICAL CHARACTERIZATION OF
CARBON PHENOLIC PREPREG MATERIALS

NAS8-36298

COPY #21

1 April 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

FILLER, CARBON BLACK

1. SCOPE

- 1.1 Scope. This specification covers one type of thermal carbon black used with carbon and graphite reinforcements for phenolic laminating applications.

2. APPLICABLE DOCUMENTS

- 2.1 Government Documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

SPECIFICATIONS

Military

MIL-STD-129 Marking for Shipment and Storage

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

- 2.2 Non-Government Documents. Unless otherwise specified, the following documents of the latest approved issues form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (FM 5055B)	CARBON CLOTH PHENOLIC, PREIMPREGNATED
MSFC-SPEC (FM 5834)	SPUN PAN PHENOLIC CLOTH, 8.5 oz/yd ² , PREIMPREGNATED
MSFC-SPEC (FM 5064J)	GRAPHITE CLOTH PHENOLIC, PREIMPREGNATED
MSFC-SPEC (FM 5839)	SPUN PAN PHENOLIC CLOTH, 6 oz/yd ² , PREIMPREGNATED

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812.)

American Society for Testing of Materials (ASTM)

ASTM D 1506	Carbon Black - Ash Content
ASTM D 1509	Carbon Black - Heating Loss
ASTM D 1510	Carbon Black - Iodine Adsorption Number
ASTM D 1512	Carbon Black - pH Value
ASTM D 1513	Carbon Black, Pelleted - Pour Density
ASTM D 1619	Carbon Black - Sulfur Content
ASTM D 2414	Carbon Black - Dibutyl Phthalate Absorption Number
ASTM D 3037	Carbon Black - Surface Area by Nitrogen Adsorption

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has been tested and has passed the qualification inspection specified herein (see 4.3).

3.1.1 Requalification. Changes in either the raw materials used, or in the methods of manufacture, after the initial qualification testing may be cause to require requalification. Notification of changes must be made in writing to the procuring activity, prior to their occurrence, and a written reply is required before proceeding.

3.2 Material Properties. The chemical and physical properties shall be in accordance with Table I.

TABLE I. MATERIAL PROPERTIES

PROPERTY	REQUIREMENT	
	MINIMUM	MAXIMUM
Carbon Assay, %	99.0	-----
Ash Content, %	-----	0.20
pH, Units	4.0	8.5
Screen Retains, % +35 mesh	-----	0.001
+335 mesh	-----	0.10

TABLE I. MATERIAL PROPERTIES (CONT'D)

PROPERTY	MINIMUM	MAXIMUM
Toluene Extracts, %	-----	0.5
Total Alkalinity, ppm	-----	50
Heating Loss, %	-----	0.1
Density, gm/cc	1.85	1.96
Particle Size, microns	-----	2.0
Dibutyl Phthalate Absorption, ml/100 gm	31	46
Iodine Adsorption, mg/gm	5	15
Sulfur Content, %	-----	0.04
Surface Area, sq. m/gm	5	20

3.3 Visual Examination. No visual contamination or any foreign material shall be allowed.

3.3.1 Shelf Life. The material shelf life shall have no limit when the material is protected from contamination and stored at ambient temperature in the original shipping container.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspections and tests specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial facility acceptable to the procuring agency. Inspection records of the examinations and tests shall be kept complete and available for a minimum of three (3) years, or longer if designated by program requirements. The procuring agency reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test results conform to prescribed requirements.

4.2 Classification of Inspections. The inspection requirements specified herein are classified as follows:

- a. Qualification inspection (see 4.3)
- b. Quality conformance inspection (see 4.4).

- 4.3 Qualification Inspection. Material units from the first production lot shall be subjected to testing for material properties as specified in Table II and inspected for conformance to Section 5. The lots subjected to qualification inspection shall be representative of the manufacturing production from the proposed production facility. any change in process or material after initial qualification testing may be cause to require requalification (see 3.1.1).

TABLE II. QUALIFICATION INSPECTION

EXAMINATION AND TESTS	REQUIREMENT	TEST METHOD
Visual Examination	3.3 and Section 5	4.7.1
Carbon Assay	3.2	4.7.2
Ash Content	3.2	4.7.3
pH	3.2	4.7.4
Screen Retains	3.2	4.7.5
Toluene Extracts	3.2	4.7.6
Total Alkalinity	3.2	4.7.7
Heating Loss	3.2	4.7.8
Density	3.2	4.7.9
Particle Size	3.2	4.7.10
Dibutyl Phthalate Absorption	3.2	4.7.11
Iodine Adsorption	3.2	4.7.12
Sulfur Content	3.2	4.7.13
Surface Area	3.2	4.7.14

- 4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of all the examinations and tests specified in Table III performed on each lot.

TABLE III. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TEST	REQUIREMENT	TEST METHOD
Visual Examination	3.3 and Section 5	4.7.1
Carbon Assay	3.2	4.7.2
Ash Content	3.2	4.7.3
pH	3.2	4.7.4

TABLE III. QUALITY CONFORMANCE INSPECTION (CONT'D)

EXAMINATIONS AND TEST	REQUIREMENT	TEST METHOD
Screen Retains	3.2	4.7.5
Toluene Extracts	3.2	4.7.6
Total Alkalinity	3.2	4.7.7
Heating Loss	3.2	4.7.8

4.5 Sampling. Each lot of carbon black filler shall be sampled for inspection as follows.

4.5.1 Lot. A lot shall consist of material produced under essentially the same conditions, in one continuous operation and offered for inspection and testing at one time.

4.5.1.1 Lot Size. A lot of carbon black filler shall consist of not more than 10,000 lbs.

4.5.2 Sample Frequency. A sample of sufficient size to perform all the required tests specified herein shall be taken from the geometric center of the shipping package after carefully removing one-inch thickness of surface material to reach the center of the bag from the position sampled. Each lot shall be sampled on the basis of five percent of the shipping packages, with a minimum of two samples per lot.

The supplier may sample from a convenient location in the process with a frequency to be one sample per 1000 pounds at a minimum or one sample per homogeneous mixture.

4.6 Test Requirements. Quality inspection testing shall be conducted as follows:

4.6.1 Test Conditions. Unless otherwise specified, testing shall be performed under ambient conditions.

4.6.2 Test Procedures. The supplier shall not be restricted to the test methods listed herein, provided an equivalent method is used. In areas of dispute, the procedure specified in this specification shall take precedence.

4.6.3 Accept/Reject Basis. If all samples meet the requirements for the tests conducted, the lot will be accepted for these criteria. Any property whose test value falls outside the required limits will be retested in duplicate from the same sample.

- 4.6.4 Test Certification. The supplier shall furnish certificates of compliance, complete with supporting test reports, to this specification. The test reports shall include, but not be limited to the quality conformance test of 4.4.
- 4.7 Test Methods. The following test methods and procedures shall be used, unless an equivalent method is mutually agreed upon. Unless otherwise specified, all weights, volumes, temperatures and times shall be measured to the nearest specified unit or decimal. Whenever value is preceded by "approximately", a tolerance of plus or minus 20 percent shall be used.
- 4.7.1 Visual Examination. Each shipping container shall be examined visually for conformance to the requirements of Section 5. Each shipping package which is opened for sampling shall be visually inspected for contamination 3.3.
- 4.7.2 Carbon Assay. The carbon assay determination shall be accomplished using a LECO CHN-600 carbon, hydrogen, nitrogen analyzer, or equivalent as follows:
- a. Dry an adequate sample in a covered crucible for 2 hours at 125°C. Remove and desiccate to ambient temperature.
 - b. Using a funnel, place the sample (normally 0.1 grams of filler) into a tared tin capsule. Crimp the top of the capsule to prevent escape of the sample.
 - c. Place the capsule with sample back on the LB-20 balance. Determine the net sample weight by subtracting the capsule tare weight.
 - d. Enter sample identification and net sample weight.
 - e. Load the capsule, with sample, in the appropriate position.
 - f. Push the analyzer key to begin analysis at a temperature of 950°C.
 - g. At completion of analysis, calibrate using a 99.99% carbon content standard.
 - h. Report the average of 3 determinations per sample to the nearest 0.1%.

4.7.3 Ash Content. The ash content of each sample shall be determined in accordance with ASTM D 1506 and the following:

- a. Ash the filler at 1750°F for a minimum of 4 hours and check for completeness of ashing by cooling the sample in a desiccator, weighing, igniting for an additional hour, cooling in a dessicator and reweighing. When ashing is determined to be complete, calculate the ash content to the nearest .01% from the average of two individual determinations.

4.7.4 pH. The pH shall be determined in accordance with ASTM D 1512 using method A - boiling slurry.

4.7.5 Screen Retains. The screen retains shall be determined in accordance with ASTM D 1508 and the following:

Use sieves with U.S. Standard No. 35 mesh and No. 325 mesh to determine the weight percent retained on each screen.

4.7.6 Toluene Extracts. The Toluene extractable percentage of each sample shall be determined with the following:

- a. Precondition a paper thimble at $325 \pm 5^\circ\text{F}$ for 120 minutes. Weigh immediately to .001 gm (W1) and store in a desiccator with the recorded weight until ready to use.
- b. Reweigh the thimble immediately before addition of the sample (W2) so an accurate sample weight is established. Place $2.5 \pm .2$ grams of carbon black in the thimble and weigh to .001 gm (W3).
- c. Reflux using Soxhlet extraction and toluene for a minimum of 4 hours. Remove the thimble, allow the solvent to drain, and dry to a constant weight at $325 \pm 5^\circ\text{F}$ for 2 hours minimum in a forced air oven. Remove from the oven and immediately weigh to .001 gm (W4).
- d. Calculate the percentage of extractable material as follows:

$$\% \text{ extractable} = 100 \left(1 - \frac{W4 - W1}{W3 - W2} \right)$$

Where W1 = weight of dried thimble, gm.

W2 = thimble weight immediately before sample addition, gm.

W3 = thimble weight plus sample, gm.

W4 = weight of thimble plus extracted sample, gm.

e. Report the average of 3 determinations to the nearest 0.01%.

4.7.7 Total Alkalinity. Total alkalinity (Na, Ca, K, Mg, Li) shall be determined in accordance with the following:

- a. Place approximately 2 gm of each sample into a separate tared crucible, and dry in an oven at $125 \pm 5^{\circ}\text{C}$ for a minimum of 1 hours. Determine the dry specimen weight to the nearest 0.1 mg.
- b. Heat the crucible in a muffle furnace at $600 \pm 15^{\circ}\text{C}$ for 16 to 18 hours or until constant weight is achieved.
- c. Remove the crucible containing the ashed specimen, place in a desiccator and cool to room temperature.
- d. Wash down the walls of each crucible with distilled water and add approximately 5 ml concentrated hydrochloric acid to each.
- e. Bring the acid mixture to a boil, cool and transfer the contents of each to a 1 liter volumetric flask and dilute to volume with distilled water.
- f. Determine the total alkalinity by flame emission or flame atomization using the instrument manufacturers operating instructions and appropriately prepared calibration curves and standards. The total alkalinity is determined by:

$$\text{Total alkalinity (ppm)} = \text{Na} + \text{Ca} + \text{K} + \text{Mg} + \text{Li}$$

4.7.8 Heating Loss. The heating loss shall be determined in accordance with ASTM D 1509.

4.7.9 Density. The density shall be determined in accordance with the following:

- a. Precondition approximately five grams of the sample in a vacuum oven heated to 275°F for 20 to 30 minutes. Remove and desiccate until ready to use.
- b. Weigh the preconditioned sample into the tared sample cup and determine the weight to 0.1 mg.
- c. Following manufacturers instructions, determine the specimen volume using helium gas.

- d. Determine density from the following:

$$\text{Density, (gm/cc)} = \frac{\text{Weight of dried specimen, gm.}}{\text{Volume reading obtained, cc.}}$$

- 4.7.10 Particle Size. The particle size shall be determined by utilizing a direct measurement technique such as a scanning electron microscope or by the following:

- a. Weigh 2 mg of the sample into a vial and dilute with ethylene glycol to 0.1 mg/ml. Mix in an ultrasonic bath for 30 minutes.
- b. Take 2 ml of the dispersion obtained in (a), and dilute to 0.01 mg/ml. Mix in an ultrasonic bath for 30 minutes.
- c. Following manufacturers instructions, determine the average particle size utilizing a Horiba centrifugal automatic particle analyzer which determines particle size based on the principle of light absorbance of a liquid dispersion of carbon black in an ethylene glycol slurry.

- 4.7.11 Dibutyl Phthalate Absorption. The dibutyl phthalate absorption number shall be determined in accordance with ASTM D 2414.

- 4.7.12 Iodine Adsorption. The iodine adsorption number shall be determined in accordance with ASTM D 1510.

- 4.7.13 Sulfur Content. The sulfur content shall be determined in accordance with ASTM D 1619.

- 4.7.14 Surface Area. The surface area shall be determined in accordance with ASTM D 3037.

5. PREPARATION FOR DELIVERY

- 5.1 Packaging. Unless otherwise specified in the purchase document, packaging and packing of carbon black filler shall be in accordance with standard commercial practice and in conformance with federal and state regulations applicable to the type of material.

- 5.2 Marking. Marking of each package shall be made as indicated in MIL-STD-129 and shall include, but not be limited to, the following:

- a. Manufacturer's name
- b. Material name and number
- c. Lot number or identification
- d. Net weight

Each outside shipping container shall include the above listed items and, in addition, the purchase document number.

- 5.3 Shipping Documentation. One copy of the certification of compliance including support test reports shall accompany each shipment.

6. NOTES

- 6.1 Intended Use. The carbon black filler covered by this specification is intended for use in the manufacture of preimpregnated graphite and carbon cloth used in the production of rocket motor nozzle components and other structural applications.

- 6.2 Ordering Data. Purchase documents should specify the following:

- a. Title, number, and date of this specification
- b. Quality conformance or qualification lot
- c. Number of copies of inspection and test date required
- d. Special packaging, packing or shipping requirements
- e. Data retention requirements

- 6.3 Qualification. The procuring activity reserves the right to purchase only those products which have been previously tested and found to meet the requirements of this specification and which have proved satisfactory in one full scale nozzle static test. Prospective suppliers whose products have not been tested and found satisfactory are required to have their products tested in order that they may be considered eligible to be awarded contracts for the product covered by this specification.

TABLE OF CONTENTS
RESIN SPECIFICATIONS
NAS8-36298
U.S. POLYMERIC O.E. 71108

<u>Description</u>	<u>Interim No.</u>
Resin, Phenolic	(91LD)
Resin, Phenylaldehyde	(USP-39A)

NOTE: Individual pages are not numbered except in sequence
for each specification.

1 March 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

RESIN, PHENOLIC

1. SCOPE

- 1.1 Scope. This specification covers one type of phenolic resin used with carbon and graphite reinforcements for laminating applications.

2. APPLICABLE DOCUMENTS

- 2.1 Government Documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

SPECIFICATIONS

Military

MIL-R-9299	Resin, Phenolic, Laminating
MIL-STD-129	Marking for Shipment and Storage

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

- 2.2 Non-Government Documents. Unless otherwise specified, the following documents of the latest approved issues form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (FM 5055B)	CARBON CLOTH PHENOLIC, PREIMPREGNATED
MSFC-SPEC (FM 5834)	SPUN PAN PHENOLIC CLOTH, 8.5 oz/yd ² , PREIMPREGNATED

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812.)

American Society for Testing of Materials (ASTM)

ASTM D 891

Specific Gravity, Apparent, of Liquid Industrial
Chemicals, Standard Test Method for(Application for copies should be addressed to the American Society for Testing
and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has been tested and has passed the qualification inspection specified herein (see 4.2.2).

3.1.1 Regualification. Changes in either the raw materials used, or in the methods of manufacture, after the initial qualification testing may be cause to require requalification. Notification of changes must be made in writing to the procuring activity, prior to their occurrence, and a written reply is required before proceeding.

3.2 Material Properties. The chemical and physical properties shall be in accordance with Table I.

TABLE I. MATERIAL PROPERTIES

PROPERTY	REQUIREMENT		TEST METHOD
	MINIMUM	MAXIMUM	
Resin Solids, %	68.0	73.0	4.4.1
Viscosity, cps @ 77°F	600	1750	4.4.2
Specific Gravity	1.120	1.150	4.4.3
Gel Time, seconds	170	250	4.4.4
Chang's Index, ml	22.5	26.0	4.4.5
Sodium Content, ppm	----	100	4.4.6
pH	8.0	8.8	4.4.7
I.R. Baseline	----	1.05	4.4.8

3.3 Storage Life. The material shall have a storage life of six months after date of manufacture, when stored at 40°F maximum in original shipping containers, and one year after date of manufacture when stored at 0°F maximum in original shipping containers.

3.3.1 Storage Life Extension. The material shall be suitable for use for applications requiring conformance to this specification as long as the following material properties are tested immediately before use and conform to Table I.

- a. Resin solids
- b. Viscosity
- c. Gel Time
- d. Chang's Index

After receipt of the material, the procuring activity is responsible for proper storage and for retest to determine storage life extensions.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspections and tests specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial facility acceptable to the procuring agency. Inspection records of the examinations and tests shall be kept complete and available for a minimum of three (3) years, or longer if designated by program requirements. The procuring agency reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test results conform to prescribed requirements.

4.2 Quality Inspections. Quality inspection shall be performed as follows:

4.2.1 Classification of Inspection: The inspection requirements specified herein are classified as follows:

- a. Qualification inspection (see 4.2.2)
- b. Quality conformance inspection (see 4.2.3)

- 4.2.2 Qualification Inspection. Material units from the first production lot (4.2.4.1) shall be subjected to testing for material properties as specified in Table I and inspected for conformance to Section 5. The lots subjected to qualification inspection shall be representative of the manufacturing production from the proposed production facility. any change in process or material after initial qualification testing may be cause to require requalification (see 3.1.1).
- 4.2.3 Quality Conformance Inspection. Material units from each production lot (see 4.2.4) shall be subjected to testing as specified in Table I and inspected for conformance to Section 5.
- 4.2.4 Sampling. Each lot shall be sampled for inspection as follows:
- 4.2.4.1 Lot. A lot shall consist of material produced under essentially the same conditions, in one continuous operation and offered for inspection and testing at one time.
- 4.2.4.2 Sample Frequency. The resin supplier may sample from each homogeneous batch after mixing and before packaging into material units for shipping. After packaging, samples representing the lot will be taken at random from the material units based on the following:
- | | |
|----------------|--|
| 1 to 5 units | 1 sample |
| 6 to 15 units | 2 samples |
| above 15 units | minimum of 10% rounded to the nearest sample |
- Samples shall be taken from the material units only after the unit has been thoroughly mixed.
- 4.3 Test Requirements. Quality inspection testing shall be conducted as follows:
- 4.3.1 Test Conditions. Unless otherwise specified, testing shall be performed under ambient conditions.
- 4.3.2 Test Procedures. The supplier shall not be restricted to the test methods listed herein, provided an equivalent method is used. In areas of dispute, the procedure specified in this specification shall take precedence.
- 4.3.3 Accept/Reject Basis. If all samples meet the requirements for the tests conducted, the lot will be accepted for these criteria. Any property whose test value falls outside the required limits will be retested in duplicate from the same sample.

- 4.3.4 Supplier Retest. Each of the duplicate values must fall within the specification limits for acceptance. If the cause of failure can be attributed to improper preparation of the specimen of a testing machine anomaly, new tests shall be conducted on the same sample.
- 4.3.5 Procuring Agency Testing. The procuring agency shall test all material properties listed in Table I which will indicate acceptability of the resin, to provide formulation data for usage of the resin, and to provide a basis for monitoring shelf life as required. If any value fails to fall within the specification limits for acceptance, resampling is required on a 100% material unit basis. Rejection is on a material unit basis up to 25%. Above 25% rejection will subject the entire lot to rejection.
- 4.3.6 Test Certification. The supplier shall furnish certificates of compliance, complete with supporting test reports, to this specification. The test reports shall include, but not be limited to, the quality conformance test of 4.2.3 and shall include certification that the resin meets the requirements of MIL-R-9299.
- 4.4 Test Methods. The following test methods and procedures shall be used, unless an equivalent method is mutually agreed upon. Unless otherwise specified, all weights, volumes, temperatures and times shall be measured to the nearest specified unit or decimal. Whenever value is preceded by "approximately", a tolerance of plus or minus 20 percent shall be used.
- 4.4.1 Resin Solids. The resin solids content in percent shall be determined in accordance with the following:
- Use a sample size which will yield approximately 1.5 grams of total solids. Estimate the weight by using the following formula:
$$\text{Sample size grams} = \frac{150}{(\text{total solids, \%})}$$
 - Fill a 2 ml. syringe with resin, and weigh to the nearest .001 gm (W4).
 - Into an aluminum dish, weighed to the nearest .001 gm (W1), expel the approximate sample weight from the syringe into the aluminum dish. Reweigh the syringe to the nearest .001 gm (W3.).

- d. Rotate the aluminum dish to spread resin over the entire bottom of the dish. Place sample in an air circulating oven set at $275 \pm 5^{\circ}\text{F}$, for 60 ± 1 minute.
- e. Remove dish from oven and cool in a desiccator. Reweigh the dish and residue to the nearest .001 gm (W_2).
- f. Calculate resin solids as follows:

$$\text{Resin solids, wt. \%} = \left(\frac{W_2 - W_1}{W_4 - W_3} \right) \times 100$$

Where W_1 = weight of aluminum dish, gms.

W_2 = weight of dish and residue, gms.

W_3 = weight of syringe, gms, after expelling sample

W_4 = weight of syringe and sample, gms.

- g. Unless otherwise specified, report the average of three determinations to 0.1%.

4.4.2 Viscosity. The viscosity of the resin shall be determined in accordance with the following:

- a. Place the sample in a container of sufficient diameter and depth so that the rotating spindle will not be closer than one inch to the container at any point.
- b. Condition the sample in a water bath to $77 \pm 0.5^{\circ}\text{F}$.
- c. Using a Brookfield viscometer, model RVF or equivalent, select a spindle consistent with the expected viscosity and immerse the spindle into the resin, level with the notch on the spindle system.
- d. Rotate the spindle at 20 rpm for one revolution with the clutch engaged. Then, release the clutch and allow equilibrium to be established. Engage the clutch, turn off the viscometer, and read the dial to the nearest 0.1 unit when the indicator appears in the window, keeping the clutch engaged.

- e. Calculate the viscosity as follows:

Viscosity in centipoise = $R \times F$

Where R = average of two dial readings

F = manufacturer's factor based on spindle number and speed

- f. Unless otherwise specified, report the viscosity to three significant figures.

- 4.4.3 Specific Gravity. Specific gravity shall be determined in accordance with ASTM D-891 and the following:

Use method A, utilizing a calibrated hydrometer, and determine at $77 \pm 0.5^\circ\text{F}$. Report results to the nearest 0.001 unit.

- 4.4.4 Gel Time. The gel time shall be determined in accordance with the following:

- a. Preheat an oil bath or equivalent bath to $338 \pm 1.5^\circ\text{F}$, while stirring the bath continually for 5 minutes prior to start of testing.
- b. Weigh into a 13mm x 120mm test tube, 2 gms \pm 0.1 gm.
- c. Use a stirring rod constructed from a 0.06 to 0.08 inch wire with a 0.30 to 0.40 inch loop perpendicular to the stem, place the stirring rod in the test tube, and suspend the rim of the test tube 1.5 inches above the bath surface. Start a stop watch immediately upon immersion.
- d. Agitate the resin with rapid vertical strokes, not more than 1/4 inch for the first 40 seconds. Then agitate at a rate of 4 strokes every 5 seconds.
- e. The gel time endpoint is defined as the elapsed time until there is no motion of the test tube in relation to the stirrer, i.e., when the tube moves up and down with the stirrer.

NOTE: Be sure the test tube holder is loose enough to permit this.

- f. Report the average of two determinations to the nearest second.

4.4.5 Chang's Index. Chang's Index shall be determined in accordance with the following:

- a. This method is an arbitrary measure of the degree of advancement of liquid phenolic resins by titrating 25 ml. of a 3.95% to 4.0% solids solution of resin in acetone with distilled water until a cloudy endpoint is observed.
- b. Initially add 50 ± 2 ml. of acetone to a flask and add 10 ± 0.2 gms. of sample to get an approximate 10-20% solids solution.
- c. Determine the resin solids by weight difference from a 2 ml. syringe (W2), after 2 ml. of solution from 4.4.5b are discharged (W1) into a tared aluminum solids dish (W3).
- d. Devolatilize the specimen in the aluminum dish in a forced air oven at $275 \pm 5^\circ\text{F}$ for 30 minutes. Cool in a desiccator and reweigh (W4).
- e. Calculate the intermediate resin solids as follows:

$$\text{Wt. \% solids} = \left(\frac{W4 - W3}{W2 - W1} \right) \times 100$$

Where W1 = syringe after sample discharged, gm.

W2 = syringe plus intermediate sample, gm.

W3 = aluminum weighing dish, gm.

W4 = aluminum dish plus residue, gm.

- f. To determine the weight, in grams, of intermediate solution to be used, use the formula:

$$\text{Wt, intermediate solution} = \frac{160}{\% \text{ solids from 4.4.5e}}$$

Wt, acetone to dilute = 40 - weight of intermediate solution

NOTE: The object of dilution is to obtain 1.6 grams of resin solids in a 40 gram total solution for the 4.0% solids solution.

- g. On an analytical balance, introduce the weight of intermediate solution from 4.4.5f into a 125 ml. flask using a large syringe as quickly as possible to the nearest 0.001 gm. Immediately, add the weight of acetone to dilute into the flask to the nearest 0.001 gms. Stopper the flask and swirl lightly to mix.

- h. Pipet 25 ml. of this 4% solution from 4.4.5g into another 125 ml. Erlenmeyer flask. Titrate with distilled water to the first perceptible, persistent endpoint. A strong compact light beam aids in determination of the endpoint since colloidal particles scatter the beam.
- i. Report the average number of ml. of distilled water for two determinations as Chang's Index.

4.4.6 Sodium Content. The sodium content shall be determined in accordance with the following:

- a. Using the resin solids weight % from 4.4.1, determine the amount of resin to add to a tared crucible to get two grams of resin solids as follows:

$$\text{Sample size} = \left(\frac{2}{\% \text{ resin solids}} \right) \times 100$$

- b. Weigh the sample to the nearest 0.1 mg.
- c. Devolatilize the sample in an air circulation oven at $225 \pm 5^\circ\text{F}$ for a minimum of 2 hours.
- d. Heat the crucible in a muffle furnace at $600 \pm 15^\circ\text{C}$ for 16-18 hours or until constant weight is achieved.
- e. Remove the crucible containing the ashed specimen, place in a desiccator and cool.
- f. Wash down the walls of the crucible with distilled water and add approximately 5 ml. concentrated hydrochloric acid.
- g. Bring the acid mixture to a boil, cool the crucible, transfer the contents to a 1 liter volumetric flask and dilute to volume with distilled water.
- h. Determine the sodium content of each flask by flame emission at 589 nanometers (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

4.4.7 pH. The pH shall be determined using a commercial pH meter with glass electrodes.

- a. Use a minimum of 100 ml. of sample in a 200 ml. tall form beaker to immerse the electrode.
- b. The pH meter must be standardized daily prior to use, using buffer standards of pH 4, 7 and 10. Follow manufacturers instructions for calibration.
- c. Report the average of duplicate determinations to the nearest 0.1 pH units.

4.4.8 Infrared Baseline. The infrared baseline shall be determined in accordance with the following:

- a. Using the resin solids weight % obtained in 4.4.1 or an estimate, determine the sample size to give approximately 2 gms. of resin solids.
- b. Evacuate the sample overnight at room temperature to remove any isopropanol.
- c. Add sufficient acetone to the resin residue to effect a 4 to 6% resin solids solution.
- d. Using a matched pair of 0.20mm cells, fill one with the resin solution and the other with water-free acetone.
- e. Place both cells in the holders of a commercial spectrophotometer and determine the absorbance at 12.15 microns. The resin concentration must be adjusted to produce a linear absorbance of 40 to 45% at 12.15 microns.
- f. When this has been achieved, scan from 5.0 to 16.0 microns at a rate of .54 microns per minute.
- g. Determine from the chart, the absorbance at 12.15 and 9.8 microns.
- h. Calculate the infrared baseline (IRZB) in the following manner:

$$\text{Baseline (IRZB)} = \frac{\log \text{ of absorbance @ 12.15 microns}}{\log \text{ of absorbance @ 9.8 microns}}$$

5. PREPARATION FOR DELIVERY

- 5.1 Packaging. The resin shall be shipped in a clean, dry and open head standard 5-gallon pails or 55-gallon drums as indicated on

the purchase order. The shipping container shall be so constructed as to insure acceptance by common or other carrier for safe transportation to the specified delivery location.

5.2 Marking. Marking of each package shall be as indicated in MIL-STD-129 and shall include, but not be limited to, the following:

- a. Manufacturer's name
- b. Material name and number
- c. Lot number
- d. Net and gross weight
- e. Purchase order number
- f. Store at 40°F or below

5.3 Special Shipping Requirements. Shipment shall be made in refrigerated trailers, capable of maintaining temperatures of 40°F or below.

5.4 Shipping Documentation. One copy of the certification of compliance including supporting test reports shall accompany each shipment.

6. NOTES

6.1 Intended Use. The resin covered by this specification is intended for use in the manufacture of preimpregnated carbon and graphite cloth used in the production of rocket motor nozzle components and other structural applications.

6.2 Ordering Data. Procurement documents shall specify, but not be limited to the following information:

- a. Type and size of container
- b. Place of delivery
- c. Request for three copies of test results and certifications of compliance to this specification and to MIL-R-9299, Type II. This shall be included as an item on the purchase order.

6.3 Storage. After receipt of the material, the procuring activity is responsible for storage and for retesting to determine storage life extension.

1 March 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

RESIN, PHENYLALDEHYDE

1. SCOPE

- 1.1 Scope. This specification covers one type of phenolic resin used with carbon and graphite reinforcements for laminating applications.

2. APPLICABLE DOCUMENTS

- 2.1 Government Documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

SPECIFICATIONS

Military

MIL-R-9299	Resin, Phenolic, Laminating
MIL-STD-129	Marking for Shipment and Storage

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

- 2.2 Non-Government Documents. Unless otherwise specified, the following documents of the latest approved issues form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (FM 5064J)	GRAPHITE CLOTH PHENOLIC, PREIMPREGNATED
MSFC-SPEC (FM 5839)	SPUN PAN PHENOLIC CLOTH, 6 oz/yd ² , PREIMPREGNATED

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812.)

American Society for Testing of Materials (ASTM)

ASTM D 891

Specific Gravity, Apparent, of Liquid Industrial Chemicals, Standard Test Method for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has been tested and has passed the qualification inspection specified herein (see 4.2.2).

3.1.1 Requalification. Changes in either the raw materials used, or in the methods of manufacture, after the initial qualification testing may be cause to require requalification. Notification of changes must be made in writing to the procuring activity, prior to their occurrence, and a written reply is required before proceeding.

3.2 Material Properties. The chemical and physical properties shall be in accordance with Table I.

TABLE I. MATERIAL PROPERTIES

PROPERTY	REQUIREMENT		TEST METHOD
	MINIMUM	MAXIMUM	
Resin Solids, %	77.0	84.0	4.4.1
Viscosity, cps @ 77°F	10,000	20,000	4.4.2
Specific Gravity	1.140	1.200	4.4.3
Gel Time, seconds	170	300	4.4.4
Chang's Index, ml	21.0	25.0	4.4.5
Sodium Content, ppm	----	200	4.4.6
pH	7.8	8.8	4.4.7
I.R. Identification	Conform to Figure #1		4.4.8

3.3 Storage Life. The material shall have a storage life of six months after date of manufacture, when stored at 40°F maximum in original shipping containers, and one year after date of manufacture when stored at 0°F maximum in original shipping containers.

3.3.1 Storage Life Extension. The material shall be suitable for use for applications requiring conformance to this specification as long as the following material properties are tested immediately before use and conform to Table I.

- a. Resin solids
- b. Viscosity
- c. Gel Time
- d. Chang's Index

After receipt of the material, the procuring activity is responsible for proper storage and for retest to determine storage life extensions.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspections and tests specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial facility acceptable to the procuring agency. Inspection records of the examinations and tests shall be kept complete and available for a minimum of three (3) years, or longer if designated by program requirements. The procuring agency reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test results conform to prescribed requirements.

4.2 Quality Inspections. Quality inspection shall be performed as follows:

4.2.1 Classification of Inspection: The inspection requirements specified herein are classified as follows:

- a. Qualification inspection (see 4.2.2)
- b. Quality conformance inspection (see 4.2.3)

- 4.2.2 Qualification Inspection. Material units from the first production lot (4.2.4.1) shall be subjected to testing for material properties as specified in Table I and inspected for conformance to Section 5. The lots subjected to qualification inspection shall be representative of the manufacturing production from the proposed production facility. any change in process or material after initial qualification testing may be cause to require requalification (see 3.1.1).
- 4.2.3 Quality Conformance Inspection. Material units from each production lot (see 4.2.4) shall be subjected to testing as specified in Table I and inspected for conformance to Section 5.
- 4.2.4 Sampling. Each lot shall be sampled for inspection as follows:
- 4.2.4.1 Lot. A lot shall consist of material produced under essentially the same conditions, in one continuous operation and offered for inspection and testing at one time.
- 4.2.4.2 Sample Frequency. The resin supplier may sample from each homogeneous batch after mixing and before packaging into material units for shipping. After packaging, samples representing the lot will be taken at random from the material units based on the following:
- | | |
|----------------|--|
| 1 to 5 units | 1 sample |
| 6 to 15 units | 2 samples |
| above 15 units | minimum of 10% rounded to the nearest sample |
- Samples shall be taken from the material units only after the unit has been thoroughly mixed.
- 4.3 Test Requirements. Quality inspection testing shall be conducted as follows:
- 4.3.1 Test Conditions. Unless otherwise specified, testing shall be performed under ambient conditions.
- 4.3.2 Test Procedures. The supplier shall not be restricted to the test methods listed herein, provided an equivalent method is used. In areas of dispute, the procedure specified in this specification shall take precedence.
- 4.3.3 Accept/Reject Basis. If all samples meet the requirements for the tests conducted, the lot will be accepted for these criteria. Any property whose test value falls outside the required limits will be retested in duplicate from the same sample.

- 4.3.4 Supplier Retest. Each of the duplicate values must fall within the specification limits for acceptance. If the cause of failure can be attributed to improper preparation of the specimen of a testing machine anomaly, new tests shall be conducted on the same sample.
- 4.3.5 Procuring Agency Testing. The procuring agency shall test all material properties listed in Table I which will indicate acceptability of the resin, to provide formulation data for usage of the resin, and to provide a basis for monitoring shelf life as required. If any value fails to fall within the specification limits for acceptance, resampling is required on a 100% material unit basis. Rejection is on a material unit basis up to 25%. Above 25% rejection will subject the entire lot to rejection.
- 4.3.6 Test Certification. The supplier shall furnish certificates of compliance, complete with supporting test reports, to this specification. The test reports shall include, but not be limited to, the quality conformance test of 4.2.3 and shall include certification that the resin meets the requirements of MIL-R-9299.
- 4.4 Test Methods. The following test methods and procedures shall be used, unless an equivalent method is mutually agreed upon. Unless otherwise specified, all weights, volumes, temperatures and times shall be measured to the nearest specified unit or decimal. Whenever value is preceded by "approximately", a tolerance of plus or minus 20 percent shall be used.
- 4.4.1 Resin Solids. The resin solids content in percent shall be determined in accordance with the following:
- Use a sample size which will yield approximately 1.5 grams of total solids. Estimate the weight by using the following formula:
$$\text{Sample size grams} = \frac{150}{(\text{total solids, \%})}$$
 - Fill a 2 ml. syringe with resin, and weigh to the nearest .001 gm (W4).
 - Into an aluminum dish, weighed to the nearest .001 gm (W1), expel the approximate sample weight from the syringe into the aluminum dish. Reweigh the syringe to the nearest .001 gm (W3.).

- d. Rotate the aluminum dish to spread resin over the entire bottom of the dish. Place sample in an air circulating oven set at $275 \pm 5^{\circ}\text{F}$, for 60 ± 1 minute.
- e. Remove dish from oven and cool in a desiccator. Reweigh the dish and residue to the nearest .001 gm (W2).
- f. Calculate resin solids as follows:

$$\text{Resin solids, wt. \%} = \left(\frac{W2 - W1}{W4 - W3} \right) \times 100$$

Where W1 = weight of aluminum dish, gms.

W2 = weight of dish and residue, gms.

W3 = weight of syringe, gms, after expelling sample

W4 = weight of syringe and sample, gms.

- g. Unless otherwise specified, report the average of three determinations to 0.1%.

4.4.2 Viscosity. The viscosity of the resin shall be determined in accordance with the following:

- a. Place the sample in a container of sufficient diameter and depth so that the rotating spindle will not be closer than one inch to the container at any point.
- b. Condition the sample in a water bath to $77 \pm 0.5^{\circ}\text{F}$.
- c. Using a Brookfield viscometer, model RVF or equivalent, select a spindle consistent with the expected viscosity and immerse the spindle into the resin, level with the notch on the spindle system.
- d. Rotate the spindle at 20 rpm for one revolution with the clutch engaged. Then, release the clutch and allow equilibrium to be established. Engage the clutch, turn off the viscometer, and read the dial to the nearest 0.1 unit when the indicator appears in the window, keeping the clutch engaged.

- e. Calculate the viscosity as follows:

$$\text{Viscosity in centipoise} = R \times F$$

Where R = average of two dial readings

F = manufacturer's factor based on spindle number and speed

- f. Unless otherwise specified, report the viscosity to three significant figures.

4.4.3 Specific Gravity. Specific gravity shall be determined in accordance with ASTM D-891 and the following:

Use method A, utilizing a calibrated hydrometer, and determine at $77 \pm 0.5^\circ\text{F}$. Report results to the nearest 0.001 unit.

4.4.4 Gel Time. The gel time shall be determined in accordance with the following:

- a. Preheat an oil bath or equivalent bath to $338 \pm 1.5^\circ\text{F}$, while stirring the bath continually for 5 minutes prior to start of testing.
- b. Weigh into a 13mm x 120mm test tube, 2 gms \pm 0.1 gm.
- c. Use a stirring rod constructed from a 0.06 to 0.08 inch wire with a 0.30 to 0.40 inch loop perpendicular to the stem, place the stirring rod in the test tube, and suspend the rim of the test tube 1.5 inches above the bath surface. Start a stop watch immediately upon immersion.
- d. Agitate the resin with rapid vertical strokes, not more than 1/4 inch for the first 40 seconds. Then agitate at a rate of 4 strokes every 5 seconds.
- e. The gel time endpoint is defined as the elapsed time until there is no motion of the test tube in relation to the stirrer, i.e., when the tube moves up and down with the stirrer.

NOTE: Be sure the test tube holder is loose enough to permit this.

- f. Report the average of two determinations to the nearest second.

4.4.5 Chang's Index. Chang's Index shall be determined in accordance with the following:

- a. This method is an arbitrary measure of the degree of advancement of liquid phenolic resins by titrating 25 ml. of a 3.95% to 4.0% solids solution of resin in acetone with distilled water until a cloudy endpoint is observed.
- b. Initially add 50 ± 2 ml. of acetone to a flask and add 10 ± 0.2 gms. of sample to get an approximate 10-20% solids solution.
- c. Determine the resin solids by weight difference from a 2 ml. syringe (W2), after 2 ml. of solution from 4.4.5b are discharged (W1) into a tared aluminum solids dish (W3).
- d. Devolatize the specimen in the aluminum dish in a forced air oven at $275 \pm 5^\circ\text{F}$ for 30 minutes. Cool in a desiccator and reweigh (W4).
- e. Calculate the intermediate resin solids as follows:

$$\text{Wt. \% solids} = \left(\frac{W4 - W3}{W2 - W1} \right) \times 100$$

Where W1 = syringe after sample discharged, gm.

W2 = syringe plus intermediate sample, gm.

W3 = aluminum weighing dish, gm.

W4 = aluminum dish plus residue, gm.

- f. To determine the weight, in grams, of intermediate solution to be used, use the formula:

$$\text{Wt, intermediate solution} = \frac{160}{\% \text{ solids from 4.4.5e}}$$

Wt, acetone to dilute = 40 - weight of intermediate solution

NOTE: The object of dilution is to obtain 1.6 grams of resin solids in a 40 gram total solution for the 4.0% solids solution.

- g. On an analytical balance, introduce the weight of intermediate solution from 4.4.5f into a 125 ml. flask using a large syringe as quickly as possible to the nearest 0.001 gm. Immediately, add the weight of acetone to dilute into the flask to the nearest 0.001 gms. Stopper the flask and swirl lightly to mix.

- h. Pipet 25 ml. of this 4% solution from 4.4.5g into another 125 ml. Erlenmeyer flask. Titrate with distilled water to the first perceptible, persistent endpoint. A strong compact light beam aids in determination of the endpoint since colloidal particles scatter the beam.
- i. Report the average number of ml. of distilled water for two determinations as Chang's Index.

4.4.6 Sodium Content. The sodium content shall be determined in accordance with the following:

- a. Using the resin solids weight % from 4.4.1, determine the amount of resin to add to a tared crucible to get two grams of resin solids as follows:

$$\text{Sample size} = \left(\frac{2}{\% \text{ resin solids}} \right) \times 100$$

- b. Weigh the sample to the nearest 0.1 mg.
- c. Devolatilize the sample in an air circulation oven at $225 \pm 5^\circ\text{F}$ for a minimum of 2 hours.
- d. Heat the crucible in a muffle furnace at $600 \pm 15^\circ\text{C}$ for 16-18 hours or until constant weight is achieved.
- e. Remove the crucible containing the ashed specimen, place in a desiccator and cool.
- f. Wash down the walls of the crucible with distilled water and add approximately 5 ml. concentrated hydrochloric acid.
- g. Bring the acid mixture to a boil, cool the crucible, transfer the contents to a 1 liter volumetric flask and dilute to volume with distilled water.
- h. Determine the sodium content of each flask by flame emission at 589 nanometers (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

4.4.7 pH. The pH shall be determined using a commercial pH meter with glass electrodes.

- a. Use a minimum of 100 ml. of sample in a 200 ml. tall form beaker to immerse the electrode.
- b. The pH meter must be standardized daily prior to use, using buffer standards of pH 4, 7 and 10. Follow manufacturers instructions for calibration.
- c. Report the average of duplicate determinations to the nearest 0.1 pH units.

4.4.8 Infrared Identification. The infrared identification shall be determined in accordance with the following:

- a. Sufficient sample shall be taken to provide 2 to 4 grams of resin solids.
- b. Thin the resin to approximately 10% solids using acetone. If the sample is cloudy, filter with S&S No. 589 filter paper or centrifuge.
- c. Place several drops of the solution on a clean dry NaCl plate to produce an even film. Place the plate in a dead air oven maintained at $100^{\circ} \pm 5^{\circ}\text{C}$ for 10 ± 1 minutes or under a heat lamp. The film should be uniform without any bare areas.
- d. Cool the NaCl plate before mounting. Mount the plate and manually check the spectrum. If there are bands which are too intense, cast a new film. If the film is not thick enough, add more diluted resin sample and devolatilize. There should be several maximum absorption bands in the 5 to 10% transmittance area.
- e. When proper film thickness has been achieved, scan the entire spectrum at 0.54 microns per minute.
- f. Compare the spectrum obtained from the sample with the standard identification spectrum attached as Figure #1. The characteristic solvent absorption bands shall not be considered foreign absorption peaks. Foreign absorption peaks indicate the presence of impurities, and could be cause for rejection. If the spectrum is qualitatively the same as Figure #1, the sample is acceptable.

5. PREPARATION FOR DELIVERY

- 5.1 Packaging. The resin shall be shipped in a clean, dry and open head standard 5-gallon pails or 55-gallon drums as indicated on

the purchase order. The shipping container shall be so constructed as to insure acceptance by common or other carrier for safe transportation to the specified delivery location.

5.2 Marking. Marking of each package shall be as indicated in MIL-STD-129 and shall include, but not be limited to, the following:

- a. Manufacturer's name
- b. Material name and number
- c. Lot number
- d. Net and gross weight
- e. Purchase order number
- f. Store at 40°F or below

5.3 Special Shipping Requirements. Shipment shall be made in refrigerated trailers, capable of maintaining temperatures of 40°F or below.

5.4 Shipping Documentation. One copy of the certification of compliance including supporting test reports shall accompany each shipment.

6. NOTES

6.1 Intended Use. The resin covered by this specification is intended for use in the manufacture of preimpregnated carbon and graphite cloth used in the production of rocket motor nozzle components and other structural applications.

6.2 Ordering Data. Procurement documents shall specify, but not be limited to the following information:

- a. Type and size of container
- b. Place of delivery
- c. Request for three copies of test results and certifications of compliance to this specification and to MIL-R-9299, Type II. This shall be included as an item on the purchase order.

6.3 Storage. After receipt of the material, the procuring activity is responsible for storage and for retesting to determine storage life extension.

ORIGINAL PAGE IS
OF POOR QUALITY

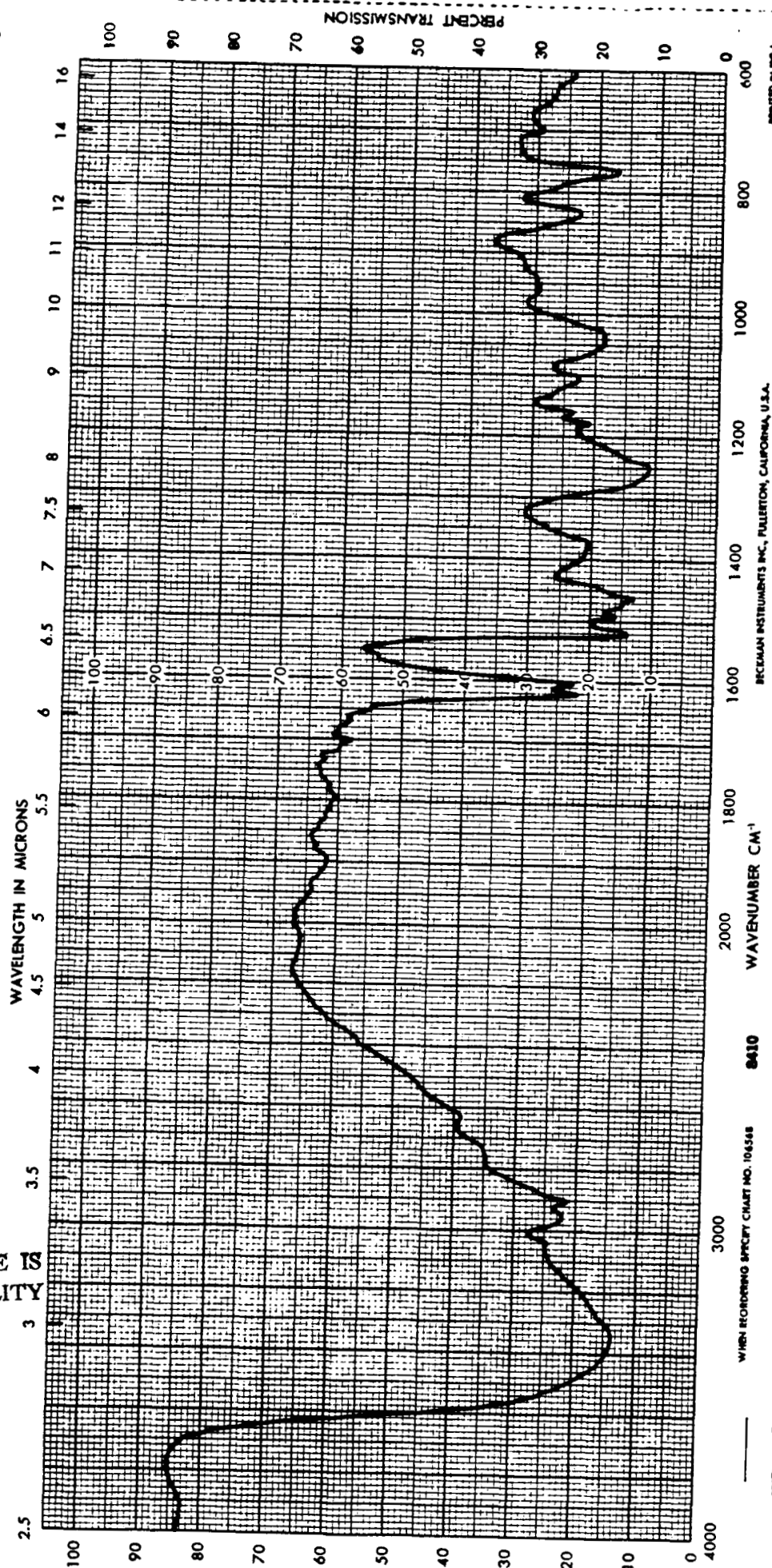


FIGURE 1. Infrared identification of resin (80% reduction)

TABLE OF CONTENTS
FABRIC SPECIFICATIONS
NAS8-36298
U.S. POLYMERIC O.E. 71108

<u>Description</u>	<u>Interim No.</u>
Reinforcement, Carbon, 1200 ppm Sodium	(CCA-3)
Reinforcement, Carbon, Spun PAN, 8.5 oz/yd ²	(SWB-8)
Reinforcement, Graphite Cloth	(WCA)
Reinforcement, Carbon, Spun PAN, 6 oz/yd ²	(PWB-6)

NOTE: Individual pages are not numbered except in sequence
for each specification.

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

REINFORCEMENT, CARBON, 1200 PPM SODIUM

1. SCOPE

- 1.1 Scope. This specification covers one type of carbon cloth reinforcement for use in solid rocket motor nozzle components.

2. APPLICABLE DOCUMENTS

- 2.1 Government documents. Not applicable.
- 2.2 Non-Government documents. Unless otherwise specified, the following non-Government documents, of the latest approved issues, form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (FM 5055B) CARBON CLOTH PHENOLIC, PREIMPREGNATED

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812)

American Society for Testing and Materials (ASTM)

ASTM C 135	True Specific Gravity of Refractory Materials by Water Immersion, Standard Test Method for
ASTM D 1682	Breaking Load and Elongation of Textile Fabrics, Standard Test Methods for
ASTM D 1777	Measuring Thickness of Textile Materials, Standard Method for
ASTM D 3775	Fabric Count of Woven Fabric, Standard Test Method for

ASTM D 3776

Mass Per Unit Area (Weight) of Woven Fabric,
Standard Test Methods for(Application for copies should be addressed to the American Society for Testing
and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has met the qualification requirements defined herein and at the usage level (see 4.3 and 6.3).

3.2 Material. The material furnished under this specification shall be a rayon-based continuous carbon filament, eight harness satin weave fabric, final processed at a temperature range of 2000 to 2850°F and suitable for use in manufacturing the preimpregnated cloth specified in MSFC-SPEC (FM 5055B).

3.2.1 Physical Properties. Physical properties of the carbon cloth reinforcement shall be in accordance with Table I.

TABLE I. PROPERTIES

MATERIAL	LIMITS	
	MINIMUM	MAXIMUM
Carbon, percent	96.0	----
Ash Content, percent	----	0.6
Moisture, percent	----	3.0
Weight, ounce per sq. yd.	7.5	9.0
Breaking Strength, lb/inch of width		
Warp Direction	20	----
Fill Direction	15	----
Specific Gravity at 25°C	1.80	1.90
Thread Count, yarns per inch		
Warp Direction	45	55
Fill Direction	45	55
Thickness, inch	0.016	0.021
Sodium Content, ppm	----	1200

- 3.3 Toxic Products and Safety. The supplier shall furnish to the procuring activity, the safety data required by U.S. Department of Labor Material Safety Data Sheet, Form OSHA-174. All the safety data applicable to the carbon reinforcement shall be supplied on Form OSHA-174 or equivalent. This requirement may be disregarded if the required data has been previously submitted.
- 3.4 Storage Life. The carbon reinforcement shall have an unlimited storage life when stored in a manner that prevents damage and contamination at warehouse ambient conditions (see 6.4).
- 3.4.1 Temperature Excursions. Not applicable.
- 3.4.2 Storage Life Extension. Not applicable.
- 3.5 Workmanship. The material shall be visibly free from flaws, impurities and other defects which would render it unsuitable for its intended use.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test methods conform to prescribed requirements.
- 4.1.1 Process Changes. The supplier shall make no changes in processing techniques or any other factors affecting the quality of the product without written approval from NASA, George C. Marshall Space Flight Center, AL 38512.
- 4.2 Classification of Inspections. Inspection and testing of the carbon reinforcement shall be classified as follows:
- a. Qualification inspection (see 4.3).
 - b. Quality conformance inspection (see 4.4).
- 4.3 Qualification Inspection. Rolls of material, which have been processed using the same procedures and facilities that will be utilized for production, shall be subjected to the quality conformance inspection specified in 4.4 using the sampling plan defined in 4.5.1.

- 4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of the examinations and tests specified in Table II using the sampling plan defined in 4.5.2.

TABLE II. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
Visual Examination	3.5 & Section 5	4.6.1
Carbon Assay	3.2.1	4.6.2
Ash Content	3.2.1	4.6.3
Moisture Content	3.2.1	4.6.4
Weight	3.2.1	4.6.4
Breaking Strength	3.2.1	4.6.5
Specific Gravity	3.2.1	4.6.6
Thread Count	3.2.1	4.6.7
Thickness	3.2.1	4.6.8
Sodium Content	3.2.1	4.6.9

4.5 Sampling

- 4.5.1 Qualification. Two rolls of material shall be processed and tested for the properties defined in Table I. Ash, carbon and sodium specimens (see 4.6.2 and 4.6.3) shall be removed from the beginning, end, and at 5 other equally spaced axial locations along the length of each roll. Each specimen shall be removed at left, center, and right locations at each axial station; 21 total test sites. Three specimens for all other properties shall be removed from the exposed end of each roll. One additional roll of material shall be randomly selected from the first production lot and subjected to the same tests as specified for the two initial rolls.
- 4.5.2 Quality Conformance. Each roll in the lot shall have three specimens removed from the exposed end and tested for carbon, ash and sodium content. For all other testing in Table II, the number of rolls to be sampled shall be in accordance with Table III. Three specimens shall be removed from the exposed end of all sampled rolls.

TABLE III. SAMPLING

NUMBER OF ROLLS IN LOT	NUMBER OF SAMPLE ROLLS TO BE SELECTED FOR TESTING
1 to 3	ALL
4 to 10	3
11 to 45	5

4.5.3 Lot. A lot shall consist of that quantity of material which is manufactured at one time, using the same process, and under the same conditions of manufacture. A lot shall not consist of more than 2500 pounds. Individual rolls shall not exceed 125 pounds.

4.5.4 Accept-Reject Criteria. The lot of material (qualification or quality conformance) shall be acceptable provided all individual values for ash, carbon and sodium content, and the average values for all other properties for each roll selected meet the requirements of Table I. Failure to meet ash, carbon, or sodium content requirements within one roll shall reject that roll.

4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified in the test or procedure description, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20% shall be used.

4.6.1 Visual Examination. All rolls shall be examined visually for conformance to 3.5. All containers shall be visually examined for conformance to the requirements of Section 5.

4.6.2 Carbon Assay Determination. The carbon assay determination shall be accomplished using a LECO CHN-600 Carbon, Hydrogen, Nitrogen Analyzer, or equivalent, as follows:

- a. Cut a specimen, 7" along the warp by 3" along the fill from the pick side, center and plain side of the fabric sample to be tested.
- b. Place the specimen in a converting oven and dry at 125 + 5°C for a minimum of 2 hours. Remove and desiccate until ready to proceed.
- c. Fold the specimen in half along the 7" side and then in thirds to yield six layers of material.
- d. With a sharp No. 1 stopper borer, cut the cloth through the six layers at least six times over the width of the folded sample.
- e. Insert the borer into the top of a tared tin capsule and use the bore cleaning tool to push out the material.
- f. Crimp the top of the capsule with forceps as the borer is drawn out to prevent escape of the sample material.
- g. Place the capsule, with sample, back on the LB-20 balance to determine net sample weight, by subtracting the capsule tare weight.
- h. Place capsule in the analyzer sample chamber, and enter sample I.D. and weight.
- i. Push the analyze key to begin analysis.
- j. When the analysis is complete, the results will be displayed and a print-out will occur.

4.6.3 Ash Content. The ash content determination shall be performed as follows:

- a. Take an approximately 2-gram, squared-shaped sample of fabric from each specified roll location and cut into 1/4 to 1/2 inch pieces.
- b. Place each specimen in tared crucibles which have been preconditioned at 600°C for one hour minimum and desiccated until used.
- c. Determine the raw sample weight to the nearest 0.1 mg.

- d. Dry in a circulating oven at $125 \pm 5^{\circ}\text{C}$ for 120 ± 10 minutes. Remove crucible and cool to room temperature in a desiccator.
- e. Determine dry sample weight to the nearest 0.1 mg.
- f. Heat the crucible in a muffle furnace at $600 \pm 15^{\circ}\text{C}$ for 16-18 hours or until constant weight is achieved.
- g. Remove the crucible containing the ashed specimen, place in a desiccator to cool and determine the ash weight to the nearest 0.1 mg.
- h. Calculate the percent ash as follows:

$$\text{Percent ash} = \frac{\text{ash weight}}{\text{dry sample weight}} \times 100$$
- i. Retain the ashed specimen for determination of sodium content.

4.6.4 Weight and Moisture. The weight of the material shall be determined in accordance with ASTM D 3776 and the moisture determined as follows:

- a. After determining the specimen weight, dry the specimen in a convection oven at $125 \pm 5^{\circ}\text{C}$ for a minimum of 120 ± 5 minutes. Remove and desiccate to room temperature.
- b. Calculate the percent moisture as follows:

$$\text{Moisture, \%} = \frac{(\text{raw sample weight} - \text{dry sample weight})}{(\text{raw sample weight})} \times 100$$

4.6.5 Breaking Strength. The breaking strength test shall be conducted as specified in ASTM D 1682.

4.6.6 Specific Gravity. The test for specific gravity shall be conducted in accordance with ASTM C 135.

4.6.7 Thread Count. The thread count of the material shall be determined in accordance with ASTM D 3775.

4.6.8 Thickness. The thickness (5 psi) shall be determined in accordance with ASTM D 1777.

4.6.9 Sodium Content. The sodium content determination shall be in accordance with the following:

- a. Obtain ash sample from 4.6.3.
- b. Wash down the walls of each crucible with distilled or laboratory grade deionized water and add approximately 5 milliliters (ml) concentrated hydrochloric acid to each.

- c. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a 200 ml. volumetric flask and dilute to volume with distilled water.
- d. Determine the sodium content of each flask by flame emission at 589 nanometres (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 2.0 and 6.0 micrograms per milliliter.
- e. Report sodium content to the nearest 10 parts per million (ppm).

5. PREPARATION FOR DELIVERY

- 5.1 Packaging and Packing. Unless otherwise specified in the purchase document, packaging and packing of carbon reinforcement shall be in accordance with standard commercial practice and in conformance with federal and state regulations applicable to the type of material. Containers in the same shipment shall be of the same size and of such construction and materials that the carbon reinforcement will be adequately protected against loss and contamination.
- 5.2 Marking. Unless otherwise specified in the purchase document, each container shall be marked for identification and shipment and shall include the following:
 - a. Supplier lot number and material name
 - b. Purchase document number
 - c. Roll identification number
 - d. Net weight of roll
 - e. This specification number and revision letter

Mark each core with supplier name, lot number, and material name.

6. NOTES

- 6.1 Intended Use. The carbon reinforcement covered by this specification is intended for use in the manufacture of carbon cloth phenolic for reinforced plastic nozzle components.
- 6.2 Ordering Data. Purchase documents should specify the following:
 - a. Title, number, and date of this specification
 - b. Quality conformance or qualification lot
 - c. Number of copies of inspection and test data required

- d. Special packaging, packing or shipping requirements
- e. Data retention requirements.

6.3 Usage Level Qualification Requirements. Material which has passed all qualification requirements defined in this specification and used in carbon cloth phenolic prepreg material shall be qualified for SRM flight nozzles after successful test in one full scale SRM static test nozzle.

6.4 Warehouse Ambient. Condition of temperature and relative humidity experienced by the material when stored in an enclosed warehouse.

1 February 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

REINFORCEMENT, CARBON, SPUN PAN, 8.5 oz/yd²

1. SCOPE

1.1 Scope. This specification covers one type of carbon cloth reinforcement for use in solid rocket motor nozzle components.

2. APPLICABLE DOCUMENTS

2.1 Government documents. Not applicable.

2.2 Non-Government documents. Unless otherwise specified, the following non-Government documents, of the latest approved issues, form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (FM 5834) SPUN PAN PHENOLIC CLOTH,
8.5 oz/yd², PREIMPREGNATED

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812)

American Society for Testing and Materials (ASTM)

ASTM C 135	True Specific Gravity of Refractory Materials by Water Immersion, Standard Test Method for
ASTM D 1682	Breaking Load and Elongation of Textile Fabrics, Standard Test Methods for
ASTM D 1777	Measuring Thickness of Textile Materials, Standard Method for
ASTM D 3775	Fabric Count of Woven Fabric, Standard Test Method for

ASTM D 3776

Mass Per Unit Area (Weight) of Woven Fabric,
Standard Test Methods for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has met the qualification requirements defined herein and at the usage level (see 4.3 and 6.3).

3.2 Material. The material furnished under this specification shall be a PAN-based spun carbon filament, eight harness satin weave fabric, final processed at a temperature range of 2600 to 3400°F and suitable for use in manufacturing the preimpregnated cloth specified in MSFC-SPEC (FM 5834).

3.2.1 Physical Properties. Physical properties of the carbon cloth reinforcement shall be in accordance with Table I.

TABLE I. PROPERTIES

MATERIAL	LIMITS	
	MINIMUM	MAXIMUM
Carbon, percent	99.0	----
Ash Content, percent	----	0.5
Moisture, percent	----	1.0
Weight, ounce per sq. yd.	7.0	11.0
Breaking Strength, lb/inch of width		
Warp Direction	20	----
Fill Direction	15	----
Specific Gravity at 25°C	1.60	2.10
Thread Count, yarns per inch		
Warp Direction	30	45
Fill Direction	30	45
Thickness, inch	0.025	0.040
Sodium Content, ppm	----	100

- 3.3 Toxic Products and Safety. The supplier shall furnish to the procuring activity, the safety data required by U.S. Department of Labor Material Safety Data Sheet, Form OSHA-174. All the safety data applicable to the carbon reinforcement shall be supplied on Form OSHA-174 or equivalent. This requirement may be disregarded if the required data has been previously submitted.
- 3.4 Storage Life. The carbon reinforcement shall have an unlimited storage life when stored in a manner that prevents damage and contamination at warehouse ambient conditions (see 6.4).
- 3.4.1 Temperature Excursions. Not applicable.
- 3.4.2 Storage Life Extension. Not applicable.
- 3.5 Workmanship. The material shall be visibly free from flaws, impurities and other defects which would render it unsuitable for its intended use.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test methods conform to prescribed requirements.
- 4.1.1 Process Changes. The supplier shall make no changes in processing techniques or any other factors affecting the quality of the product without written approval from NASA, George C. Marshall Space Flight Center, AL 38512.
- 4.2 Classification of Inspections. Inspection and testing of the carbon reinforcement shall be classified as follows:
- a. Qualification inspection (see 4.3).
 - b. Quality conformance inspection (see 4.4).
- 4.3 Qualification Inspection. Rolls of material, which have been processed using the same procedures and facilities that will be utilized for production, shall be subjected to the quality conformance inspection specified in 4.4 using the sampling plan defined in 4.5.1.

- 4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of the examinations and tests specified in Table II using the sampling plan defined in 4.5.2.

TABLE II. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
Visual Examination	3.5 & Section 5	4.6.1
Carbon Assay	3.2.1	4.6.2
Ash Content	3.2.1	4.6.3
Moisture Content	3.2.1	4.6.4
Weight	3.2.1	4.6.4
Breaking Strength	3.2.1	4.6.5
Specific Gravity	3.2.1	4.6.6
Thread Count	3.2.1	4.6.7
Thickness	3.2.1	4.6.8
Sodium Content	3.2.1	4.6.9

4.5 Sampling

- 4.5.1 Qualification. Two rolls of material shall be processed and tested for the properties defined in Table I. Ash, carbon and sodium specimens (see 4.6.2 and 4.6.3) shall be removed from the beginning, end, and at 5 other equally spaced axial locations along the length of each roll. Each specimen shall be removed at left, center, and right locations at each axial station; 21 total test sites. Three specimens for all other properties shall be removed from the exposed end of each roll. One additional roll of material shall be randomly selected from the first production lot and subjected to the same tests as specified for the two initial rolls.
- 4.5.2 Quality Conformance. Each roll in the lot shall have three specimens removed from the exposed end and tested for carbon, ash and sodium content. For all other testing in Table II, the number of rolls to be sampled shall be in accordance with Table III. Three specimens shall be removed from the exposed end of all sampled rolls.

TABLE III. SAMPLING

NUMBER OF ROLLS IN LOT	NUMBER OF SAMPLE ROLLS TO BE SELECTED FOR TESTING
1 to 3	ALL
4 to 10	3
11 to 45	5

- 4.5.3 Lot. A lot shall consist of that quantity of material which is manufactured at one time, using the same process, and under the same conditions of manufacture. A lot shall not consist of more than 2500 pounds. Individual rolls shall not exceed 125 pounds.
- 4.5.4 Accept-Reject Criteria. The lot of material (qualification or quality conformance) shall be acceptable provided all individual values for ash, carbon and sodium content, and the average values for all other properties for each roll selected meet the requirements of Table I. Failure to meet ash, carbon, or sodium content requirements within one roll shall reject that roll.
- 4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified in the test or procedure description, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20% shall be used.
- 4.6.1 Visual Examination. All rolls shall be examined visually for conformance to 3.5. All containers shall be visually examined for conformance to the requirements of Section 5.

4.6.2 Carbon Assay Determination. The carbon assay determination shall be accomplished using a LECO CHN-600 Carbon, Hydrogen, Nitrogen Analyzer, or equivalent, as follows:

- a. Cut a specimen, 7" along the warp by 3" along the fill from the pick side, center and plain side of the fabric sample to be tested.
- b. Place the specimen in a converting oven and dry at $125 \pm 5^{\circ}\text{C}$ for a minimum of 2 hours. Remove and desiccate until ready to proceed.
- c. Fold the specimen in half along the 7" side and then in thirds to yield six layers of material.
- d. With a sharp No. 1 stopper borer, cut the cloth through the six layers at least six times over the width of the folded sample.
- e. Insert the borer into the top of a tared tin capsule and use the bore cleaning tool to push out the material.
- f. Crimp the top of the capsule with forceps as the borer is drawn out to prevent escape of the sample material.
- g. Place the capsule, with sample, back on the LB-20 balance to determine net sample weight, by subtracting the capsule tare weight.
- h. Place capsule in the analyzer sample chamber, and enter sample I.D. and weight.
- i. Push the analyze key to begin analysis.
- j. When the analysis is complete, the results will be displayed and a print-out will occur.

4.6.3 Ash Content. The ash content determination shall be performed as follows:

- a. Take an approximately 2-gram, squared-shaped sample of fabric from each specified roll location and cut into 1/4 to 1/2 inch pieces.
- b. Place each specimen in tared crucibles which have been preconditioned at 600°C for one hour minimum and desiccated until used.
- c. Determine the raw sample weight to the nearest 0.1 mg.

- d. Dry in a circulating oven at $125 \pm 5^{\circ}\text{C}$ for 120 ± 10 minutes. Remove crucible and cool to room temperature in a desiccator.
- e. Determine dry sample weight to the nearest 0.1 mg.
- f. Heat the crucible in a muffle furnace at $600 \pm 15^{\circ}\text{C}$ for 16-18 hours or until constant weight is achieved.
- g. Remove the crucible containing the ashed specimen, place in a desiccator to cool and determine the ash weight to the nearest 0.1 mg.
- h. Calculate the percent ash as follows:

$$\text{Percent ash} = \frac{\text{ash weight}}{\text{dry sample weight}} \times 100$$
- i. Retain the ashed specimen for determination of sodium content.

4.6.4 Weight and Moisture. The weight of the material shall be determined in accordance with ASTM D 3776 and the moisture determined as follows:

- a. After determining the specimen weight, dry the specimen in a convection oven at $125 \pm 5^{\circ}\text{C}$ for a minimum of 120 ± 5 minutes. Remove and desiccate to room temperature.
- b. Calculate the percent moisture as follows:

$$\text{Moisture, \%} = \frac{(\text{raw sample weight} - \text{dry sample weight})}{(\text{raw sample weight})} \times 100$$

4.6.5 Breaking Strength. The breaking strength test shall be conducted as specified in ASTM D 1682.

4.6.6 Specific Gravity. The test for specific gravity shall be conducted in accordance with ASTM C 135.

4.6.7 Thread Count. The thread count of the material shall be determined in accordance with ASTM D 3775.

4.6.8 Thickness. The thickness (5 psi) shall be determined in accordance with ASTM D 1777.

4.6.9 Sodium Content. The sodium content determination shall be in accordance with the following:

- a. Obtain ash sample from 4.6.3.
- b. Wash down the walls of each crucible with distilled or laboratory grade deionized water and add approximately 5 milliliters (ml) concentrated hydrochloric acid to each.

- c. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a one liter volumetric flask and dilute to volume with distilled water.
- d. Determine the sodium content of each flask by flame emission at 589 nanometres (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.
- e. Report sodium content to the nearest 1 part per million (ppm).

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. Unless otherwise specified in the purchase document, packaging and packing of carbon reinforcement shall be in accordance with standard commercial practice and in conformance with federal and state regulations applicable to the type of material. Containers in the same shipment shall be of the same size and of such construction and materials that the carbon reinforcement will be adequately protected against loss and contamination.

5.2 Marking. Unless otherwise specified in the purchase document, each container shall be marked for identification and shipment and shall include the following:

- a. Supplier lot number and material name
- b. Purchase document number
- c. Roll identification number
- d. Net weight of roll
- e. This specification number and revision letter

Mark each core with supplier name, lot number, and material name.

6. NOTES

6.1 Intended Use. The carbon reinforcement covered by this specification is intended for use in the manufacture of carbon cloth phenolic for reinforced plastic nozzle components.

6.2 Ordering Data. Purchase documents should specify the following:

- a. Title, number, and date of this specification
- b. Quality conformance or qualification lot

- c. Number of copies of inspection and test data required
- d. Special packaging, packing or shipping requirements
- e. Data retention requirements.

6.3 Usage Level Qualification Requirements. Material which has passed all qualification requirements defined in this specification and used in carbon cloth phenolic prepreg material shall be qualified for SRM flight nozzles after successful test in one full scale SRM static test nozzle.

6.4 Warehouse Ambient. Condition of temperature and relative humidity experienced by the material when stored in an enclosed warehouse.

1 February 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

REINFORCEMENT, GRAPHITE CLOTH

1. SCOPE

- 1.1 Scope. This specification covers one type of graphite cloth reinforcement for use in solid rocket motor nozzle components.

2. APPLICABLE DOCUMENTS

- 2.1 Government documents. Not applicable.
- 2.2 Non-Government documents. Unless otherwise specified, the following non-Government documents, of the latest approved issues, form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (FM 5064J) GRAPHITE CLOTH PHENOLIC, PREIMPREGNATED

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812)

American Society for Testing and Materials (ASTM)

ASTM C 135	True Specific Gravity of Refractory Materials by Water Immersion, Standard Test Method for
ASTM D 1682	Breaking Load and Elongation of Textile Fabrics, Standard Test Methods for
ASTM D 1777	Measuring Thickness of Textile Materials, Standard Method for
ASTM D 3775	Fabric Count of Woven Fabric, Standard Test Method for

ASTM D 3776

Mass Per Unit Area (Weight) of Woven Fabric,
Standard Test Methods for(Application for copies should be addressed to the American Society for Testing
and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has met the qualification requirements defined herein and at the usage level (see 4.3 and 6.3).

3.2 Material. The material furnished under this specification shall be a rayon-based graphite plain weave fabric, final processed at a temperature range of 4700-5200°F and suitable for use in manufacturing the preimpregnated cloth specified in MSFC-SPEC (FM 5064J).

3.2.1 Physical Properties. Physical properties of the graphite cloth reinforcement shall be in accordance with Table I.

TABLE I. PROPERTIES

MATERIAL	LIMITS	
	MINIMUM	MAXIMUM
Carbon, percent	99.0	----
Ash Content, percent	----	0.5
Moisture, percent	----	1.0
Weight, ounce per sq. yd.	6.5	8.5
Breaking Strength, lb/inch of width		
Warp Direction	10	----
Fill Direction	10	----
Specific Gravity at 25°C	1.38	1.50
Thread Count, yarns per inch		
Warp Direction	22	30
Fill Direction	20	28
Thickness, inch	0.017	0.027
Sodium Content, ppm	----	100

- 3.3 Toxic Products and Safety. The supplier shall furnish to the procuring activity, the safety data required by U.S. Department of Labor Material Safety Data Sheet, Form OSHA-174. All the safety data applicable to the graphite reinforcement shall be supplied on Form OSHA-174 or equivalent. This requirement may be disregarded if the required data has been previously submitted.
- 3.4 Storage Life. The graphite reinforcement shall have an unlimited storage life when stored in a manner that prevents damage and contamination at warehouse ambient conditions (see 6.4).
- 3.4.1 Temperature Excursions. Not applicable.
- 3.4.2 Storage Life Extension. Not applicable.
- 3.5 Workmanship. The material shall be visibly free from flaws, impurities and other defects which would render it unsuitable for its intended use.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test methods conform to prescribed requirements.
- 4.1.1 Process Changes. The supplier shall make no changes in processing techniques or any other factors affecting the quality of the product without written approval from NASA, George C. Marshall Space Flight Center, AL 38512.
- 4.2 Classification of Inspections. Inspection and testing of the graphite reinforcement shall be classified as follows:
- a. Qualification inspection (see 4.3).
 - b. Quality conformance inspection (see 4.4).
- 4.3 Qualification Inspection. Rolls of material, which have been processed using the same procedures and facilities that will be utilized for production, shall be subjected to the quality conformance inspection specified in 4.4 using the sampling plan defined in 4.5.1.

- 4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of the examinations and tests specified in Table II using the sampling plan defined in 4.5.2.

TABLE II. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
Visual Examination	3.5 & Section 5	4.6.1
Carbon Assay	3.2.1	4.6.2
Ash Content	3.2.1	4.6.3
Moisture Content	3.2.1	4.6.4
Weight	3.2.1	4.6.4
Breaking Strength	3.2.1	4.6.5
Specific Gravity	3.2.1	4.6.6
Thread Count	3.2.1	4.6.7
Thickness	3.2.1	4.6.8
Sodium Content	3.2.1	4.6.9

4.5 Sampling

- 4.5.1 Qualification. Two rolls of material shall be processed and tested for the properties defined in Table I. Ash, carbon and sodium specimens (see 4.6.2 and 4.6.3) shall be removed from the beginning, end, and at 5 other equally spaced axial locations along the length of each roll. Each specimen shall be removed at left, center, and right locations at each axial station; 21 total test sites. Three specimens for all other properties shall be removed from the exposed end of each roll. One additional roll of material shall be randomly selected from the first production lot and subjected to the same tests as specified for the two initial rolls.
- 4.5.2 Quality Conformance. Each roll in the lot shall have three specimens removed from the exposed end and tested for carbon, ash and sodium content. For all other testing in Table II, the number of rolls to be sampled shall be in accordance with Table III. Three specimens shall be removed from the exposed end of all sampled rolls.

TABLE III. SAMPLING

NUMBER OF ROLLS IN LOT	NUMBER OF SAMPLE ROLLS TO BE SELECTED FOR TESTING
1 to 3	ALL
4 to 10	3
11 to 45	5

4.5.3 Lot. A lot shall consist of that quantity of material which is manufactured at one time, using the same process, and under the same conditions of manufacture. A lot shall not consist of more than 2500 pounds. Individual rolls shall not exceed 125 pounds.

4.5.4 Accept-Reject Criteria. The lot of material (qualification or quality conformance) shall be acceptable provided all individual values for ash, carbon and sodium content, and the average values for all other properties for each roll selected meet the requirements of Table I. Failure to meet ash, carbon, or sodium content requirements within one roll shall reject that roll.

4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified in the test or procedure description, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20% shall be used.

4.6.1 Visual Examination. All rolls shall be examined visually for conformance to 3.5. All containers shall be visually examined for conformance to the requirements of Section 5.

4.6.2 Carbon Assay Determination. The carbon assay determination shall be accomplished using a LECO CHN-600 Carbon, Hydrogen, Nitrogen Analyzer, or equivalent, as follows:

- a. Cut a specimen, 7" along the warp by 3" along the fill from the pick side, center and plain side of the fabric sample to be tested.
- b. Place the specimen in a converting oven and dry at $125 \pm 5^{\circ}\text{C}$ for a minimum of 2 hours. Remove and desiccate until ready to proceed.
- c. Fold the specimen in half along the 7" side and then in thirds to yield six layers of material.
- d. With a sharp No. 1 stopper borer, cut the cloth through the six layers at least six times over the width of the folded sample.
- e. Insert the borer into the top of a tared tin capsule and use the bore cleaning tool to push out the material.
- f. Crimp the top of the capsule with forceps as the borer is drawn out to prevent escape of the sample material.
- g. Place the capsule, with sample, back on the LB-20 balance to determine net sample weight, by subtracting the capsule tare weight.
- h. Place capsule in the analyzer sample chamber, and enter sample I.D. and weight.
- i. Push the analyze key to begin analysis.
- j. When the analysis is complete, the results will be displayed and a print-out will occur.

4.6.3 Ash Content. The ash content determination shall be performed as follows:

- a. Take an approximately 2-gram, squared-shaped sample of fabric from each specified roll location and cut into 1/4 to 1/2 inch pieces.
- b. Place each specimen in tared crucibles which have been preconditioned at 600°C for one hour minimum and desiccated until used.
- c. Determine the raw sample weight to the nearest 0.1 mg.

- d. Dry in a circulating oven at $125 \pm 5^{\circ}\text{C}$ for 120 ± 10 minutes. Remove crucible and cool to room temperature in a desiccator.
- e. Determine dry sample weight to the nearest 0.1 mg.
- f. Heat the crucible in a muffle furnace at $600 \pm 15^{\circ}\text{C}$ for 16-18 hours or until constant weight is achieved.
- g. Remove the crucible containing the ashed specimen, place in a desiccator to cool and determine the ash weight to the nearest 0.1 mg.
- h. Calculate the percent ash as follows:

$$\text{Percent ash} = \frac{\text{ash weight}}{\text{dry sample weight}} \times 100$$
- i. Retain the ashed specimen for determination of sodium content.

4.6.4 Weight and Moisture. The weight of the material shall be determined in accordance with ASTM D 3776 and the moisture determined as follows:

- a. After determining the specimen weight, dry the specimen in a convection oven at $125 \pm 5^{\circ}\text{C}$ for a minimum of 120 ± 5 minutes. Remove and desiccate to room temperature.
- b. Calculate the percent moisture as follows:

$$\text{Moisture, \%} = \frac{(\text{raw sample weight} - \text{dry sample weight})}{(\text{raw sample weight})} \times 100$$

4.6.5 Breaking Strength. The breaking strength test shall be conducted as specified in ASTM D 1682.

4.6.6 Specific Gravity. The test for specific gravity shall be conducted in accordance with ASTM C 135.

4.6.7 Thread Count. The thread count of the material shall be determined in accordance with ASTM D 3775.

4.6.8 Thickness. The thickness (5 psi) shall be determined in accordance with ASTM D 1777.

4.6.9 Sodium Content. The sodium content determination shall be in accordance with the following:

- a. Obtain ash sample from 4.6.3.
- b. Wash down the walls of each crucible with distilled or laboratory grade deionized water and add approximately 5 milliliters (ml) concentrated hydrochloric acid to each.

- c. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a one liter volumetric flask and dilute to volume with distilled water.
- d. Determine the sodium content of each flask by flame emission at 589 nanometres (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.
- e. Report sodium content to the nearest 1 part per million (ppm).

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. Unless otherwise specified in the purchase document, packaging and packing of graphite reinforcement shall be in accordance with standard commercial practice and in conformance with federal and state regulations applicable to the type of material. Containers in the same shipment shall be of the same size and of such construction and materials that the graphite reinforcement will be adequately protected against loss and contamination.

5.2 Marking. Unless otherwise specified in the purchase document, each container shall be marked for identification and shipment and shall include the following:

- a. Supplier lot number and material name
- b. Purchase document number
- c. Roll identification number
- d. Net weight of roll
- e. This specification number and revision letter

Mark each core with supplier name, lot number, and material name.

6. NOTES

6.1 Intended Use. The carbon reinforcement covered by this specification is intended for use in the manufacture of graphite cloth phenolic for reinforced plastic nozzle components.

6.2 Ordering Data. Purchase documents should specify the following:

- a. Title, number, and date of this specification
- b. Quality conformance or qualification lot

- c. Number of copies of inspection and test data required
- d. Special packaging, packing or shipping requirements
- e. Data retention requirements.

6.3 Usage Level Qualification Requirements. Material which has passed all qualification requirements defined in this specification and used in graphite cloth phenolic prepreg material shall be qualified for SRM flight nozzles after successful test in one full scale SRM static test nozzle.

6.4 Warehouse Ambient. Condition of temperature and relative humidity experienced by the material when stored in an enclosed warehouse.

1 February 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

REINFORCEMENT, CARBON, SPUN PAN, 6 oz/yd²

1. SCOPE

- 1.1 Scope. This specification covers one type of carbon cloth reinforcement for use in solid rocket motor nozzle components.

2. APPLICABLE DOCUMENTS

- 2.1 Government documents. Not applicable.
- 2.2 Non-Government documents. Unless otherwise specified, the following non-Government documents, of the latest approved issues, form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (FM 5839) SPUN PAN PHENOLIC CLOTH,
6 oz/yd², PREIMPREGNATED

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812)

American Society for Testing and Materials (ASTM)

ASTM C 135	True Specific Gravity of Refractory Materials by Water Immersion, Standard Test Method for
ASTM D 1682	Breaking Load and Elongation of Textile Fabrics, Standard Test Methods for
ASTM D 1777	Measuring Thickness of Textile Materials, Standard Method for
ASTM D 3775	Fabric Count of Woven Fabric, Standard Test Method for

ASTM D 3776

Mass Per Unit Area (Weight) of Woven Fabric,
Standard Test Methods for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has met the qualification requirements defined herein and at the usage level (see 4.3 and 6.3).

3.2 Material. The material furnished under this specification shall be a PAN-based spun carbon filament, plain weave fabric, final processed at a temperature range of 2600 to 3400°F and suitable for use in manufacturing the preimpregnated cloth specified in MSFC-SPEC (FM 5839).

3.2.1 Physical Properties. Physical properties of the carbon cloth reinforcement shall be in accordance with Table I.

TABLE I. PROPERTIES

MATERIAL	LIMITS	
	MINIMUM	MAXIMUM
Carbon, percent	99.0	----
Ash Content, percent	----	0.5
Moisture, percent	----	1.0
Weight, ounce per sq. yd.	5.0	8.5
Breaking Strength, lb/inch of width		
Warp Direction	10	----
Fill Direction	10	----
Specific Gravity at 25°C	1.70	1.90
Thread Count, yarns per inch		
Warp Direction	25	35
Fill Direction	25	35
Thickness, inch	0.017	0.035
Sodium Content, ppm	----	100

3.3 Toxic Products and Safety. The supplier shall furnish to the procuring activity, the safety data required by U.S. Department of Labor Material Safety Data Sheet, Form OSHA-174. All the safety data applicable to the carbon reinforcement shall be supplied on Form OSHA-174 or equivalent. This requirement may be disregarded if the required data has been previously submitted.

3.4 Storage Life. The carbon reinforcement shall have an unlimited storage life when stored in a manner that prevents damage and contamination at warehouse ambient conditions (see 6.4).

3.4.1 Temperature Excursions. Not applicable.

3.4.2 Storage Life Extension. Not applicable.

3.5 Workmanship. The material shall be visibly free from flaws, impurities and other defects which would render it unsuitable for its intended use.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test methods conform to prescribed requirements.

4.1.1 Process Changes. The supplier shall make no changes in processing techniques or any other factors affecting the quality of the product without written approval from NASA, George C. Marshall Space Flight Center, AL 38512.

4.2 Classification of Inspections. Inspection and testing of the carbon reinforcement shall be classified as follows:

a. Qualification inspection (see 4.3).

b. Quality conformance inspection (see 4.4).

4.3 Qualification Inspection. Rolls of material, which have been processed using the same procedures and facilities that will be utilized for production, shall be subjected to the quality conformance inspection specified in 4.4 using the sampling plan defined in 4.5.1.

- 4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of the examinations and tests specified in Table II using the sampling plan defined in 4.5.2.

TABLE II. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
Visual Examination	3.5 & Section 5	4.6.1
Carbon Assay	3.2.1	4.6.2
Ash Content	3.2.1	4.6.3
Moisture Content	3.2.1	4.6.4
Weight	3.2.1	4.6.4
Breaking Strength	3.2.1	4.6.5
Specific Gravity	3.2.1	4.6.6
Thread Count	3.2.1	4.6.7
Thickness	3.2.1	4.6.8
Sodium Content	3.2.1	4.6.9

4.5 Sampling

- 4.5.1 Qualification. Two rolls of material shall be processed and tested for the properties defined in Table I. Ash, carbon and sodium specimens (see 4.6.2 and 4.6.3) shall be removed from the beginning, end, and at 5 other equally spaced axial locations along the length of each roll. Each specimen shall be removed at left, center, and right locations at each axial station; 21 total test sites. Three specimens for all other properties shall be removed from the exposed end of each roll. One additional roll of material shall be randomly selected from the first production lot and subjected to the same tests as specified for the two initial rolls.
- 4.5.2 Quality Conformance. Each roll in the lot shall have three specimens removed from the exposed end and tested for carbon, ash and sodium content. For all other testing in Table II, the number of rolls to be sampled shall be in accordance with Table III. Three specimens shall be removed from the exposed end of all sampled rolls.

TABLE III. SAMPLING

NUMBER OF ROLLS IN LOT	NUMBER OF SAMPLE ROLLS TO BE SELECTED FOR TESTING
1 to 3	ALL
4 to 10	3
11 to 45	5

4.5.3 Lot. A lot shall consist of that quantity of material which is manufactured at one time, using the same process, and under the same conditions of manufacture. A lot shall not consist of more than 2500 pounds. Individual rolls shall not exceed 125 pounds.

4.5.4 Accept-Reject Criteria. The lot of material (qualification or quality conformance) shall be acceptable provided all individual values for ash, carbon and sodium content, and the average values for all other properties for each roll selected meet the requirements of Table I. Failure to meet ash, carbon, or sodium content requirements within one roll shall reject that roll.

4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified in the test or procedure description, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20% shall be used.

4.6.1 Visual Examination. All rolls shall be examined visually for conformance to 3.5. All containers shall be visually examined for conformance to the requirements of Section 5.

4.6.2 Carbon Assay Determination. The carbon assay determination shall be accomplished using a LECO CHN-600 Carbon, Hydrogen, Nitrogen Analyzer, or equivalent, as follows:

- a. Cut a specimen, 7" along the warp by 3" along the fill from the pick side, center and plain side of the fabric sample to be tested.
- b. Place the specimen in a converting oven and dry at $125 \pm 5^{\circ}\text{C}$ for a minimum of 2 hours. Remove and desiccate until ready to proceed.
- c. Fold the specimen in half along the 7" side and then in thirds to yield six layers of material.
- d. With a sharp No. 1 stopper borer, cut the cloth through the six layers at least six times over the width of the folded sample.
- e. Insert the borer into the top of a tared tin capsule and use the bore cleaning tool to push out the material.
- f. Crimp the top of the capsule with forceps as the borer is drawn out to prevent escape of the sample material.
- g. Place the capsule, with sample, back on the LB-20 balance to determine net sample weight, by subtracting the capsule tare weight.
- h. Place capsule in the analyzer sample chamber, and enter sample I.D. and weight.
- i. Push the analyze key to begin analysis.
- j. When the analysis is complete, the results will be displayed and a print-out will occur.

4.6.3 Ash Content. The ash content determination shall be performed as follows:

- a. Take an approximately 2-gram, squared-shaped sample of fabric from each specified roll location and cut into 1/4 to 1/2 inch pieces.
- b. Place each specimen in tared crucibles which have been preconditioned at 600°C for one hour minimum and desiccated until used.
- c. Determine the raw sample weight to the nearest 0.1 mg.

- d. Dry in a circulating oven at $125 \pm 5^\circ\text{C}$ for 120 ± 10 minutes. Remove crucible and cool to room temperature in a desiccator.
- e. Determine dry sample weight to the nearest 0.1 mg.
- f. Heat the crucible in a muffle furnace at $600 \pm 15^\circ\text{C}$ for 16-18 hours or until constant weight is achieved.
- g. Remove the crucible containing the ashed specimen, place in a desiccator to cool and determine the ash weight to the nearest 0.1 mg.
- h. Calculate the percent ash as follows:

$$\text{Percent ash} = \frac{\text{ash weight}}{\text{dry sample weight}} \times 100$$
- i. Retain the ashed specimen for determination of sodium content.

4.6.4 Weight and Moisture. The weight of the material shall be determined in accordance with ASTM D 3776 and the moisture determined as follows:

- a. After determining the specimen weight, dry the specimen in a convection oven at $125 \pm 5^\circ\text{C}$ for a minimum of 120 ± 5 minutes. Remove and desiccate to room temperature.
- b. Calculate the percent moisture as follows:

$$\text{Moisture, \%} = \frac{(\text{raw sample weight} - \text{dry sample weight})}{(\text{raw sample weight})} \times 100$$

4.6.5 Breaking Strength. The breaking strength test shall be conducted as specified in ASTM D 1682.

4.6.6 Specific Gravity. The test for specific gravity shall be conducted in accordance with ASTM C 135.

4.6.7 Thread Count. The thread count of the material shall be determined in accordance with ASTM D 3775.

4.6.8 Thickness. The thickness (5 psi) shall be determined in accordance with ASTM D 1777.

4.6.9 Sodium Content. The sodium content determination shall be in accordance with the following:

- a. Obtain ash sample from 4.6.3.
- b. Wash down the walls of each crucible with distilled or laboratory grade deionized water and add approximately 5 milliliters (ml) concentrated hydrochloric acid to each.

- c. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a one liter volumetric flask and dilute to volume with distilled water.
- d. Determine the sodium content of each flask by flame emission at 589 nanometres (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.
- e. Report sodium content to the nearest 1 part per million (ppm).

5. PREPARATION FOR DELIVERY

- 5.1 Packaging and Packing. Unless otherwise specified in the purchase document, packaging and packing of carbon reinforcement shall be in accordance with standard commercial practice and in conformance with federal and state regulations applicable to the type of material. Containers in the same shipment shall be of the same size and of such construction and materials that the carbon reinforcement will be adequately protected against loss and contamination.
- 5.2 Marking. Unless otherwise specified in the purchase document, each container shall be marked for identification and shipment and shall include the following:
 - a. Supplier lot number and material name
 - b. Purchase document number
 - c. Roll identification number
 - d. Net weight of roll
 - e. This specification number and revision letter

Mark each core with supplier name, lot number, and material name.

6. NOTES

- 6.1 Intended Use. The carbon reinforcement covered by this specification is intended for use in the manufacture of carbon cloth phenolic for reinforced plastic nozzle components.
- 6.2 Ordering Data. Purchase documents should specify the following:
 - a. Title, number, and date of this specification
 - b. Quality conformance or qualification lot

- c. Number of copies of inspection and test data required
- d. Special packaging, packing or shipping requirements
- e. Data retention requirements.

6.3 Usage Level Qualification Requirements. Material which has passed all qualification requirements defined in this specification and used in carbon cloth phenolic prepreg material shall be qualified for SRM flight nozzles after successful test in one full scale SRM static test nozzle.

6.4 Warehouse Ambient. Condition of temperature and relative humidity experienced by the material when stored in an enclosed warehouse.

TABLE OF CONTENTS
PREPREG SPECIFICATIONS
NAS8-36298
U.S. POLYMERIC O.E. 71108

<u>Description</u>	<u>Interim No.</u>
Carbon Cloth Phenolic, Preimpregnated	(FM 5055B)
Spun PAN Phenolic Cloth 8.5 oz/yd ² Preimpregnated	(FM 5834)
Graphite Cloth Phenolic, Preimpregnated	(FM 5064J)
Spun PAN Phenolic Cloth 6 oz/yd ² Preimpregnated	(FM 5839)

NOTE: Individual pages are not numbered except in sequence
for each specification.

1 February 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

CARBON CLOTH PHENOLIC, PREIMPREGNATED

1. SCOPE

- 1.1 Scope. This specification covers one type of carbon cloth reinforcement preimpregnated with a carbon filled phenolic resin.

2. APPLICABLE DOCUMENTS

- 2.1 Government Documents. The following documents of the issue in effect on date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

SPECIFICATIONS

Military

MIL-B-131	Barrier Materials, Watervapor-proof, Grease-proof, Flexible, Heat-sealable
MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification
MIL-R-9299	Resin, Phenolic, Laminating

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

- 2.2 Non-Government Documents. Unless otherwise specified, the following documents of the latest approved issues form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (USP-28) Filler, Carbon Black

MSFC-SPEC (CCA-3) Reinforcement, Carbon, 1200 PPM Sodium

MSFC-SPEC (91LD) Resin, Phenolic

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812.)

American Society for Testing and Materials (ASTM)

ASTM C 177	Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate, Standard Test Method for
ASTM C 613	Resin Content of Carbon and Graphite Prepregs by Solvent Extraction, Standard Test Method for
ASTM D 638	Tensile Properties of Plastics, Standard Test Method for
ASTM D 790	Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials, Standard Test Methods for
ASTM E 831	Linear Thermal Expansion of Solid Materials by Thermodilatometry, Standard Test Method for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

- 3.1 Qualification. The material furnished under this specification shall be a product which has been tested and has passed the qualification inspection specified herein (see 4.3 and 6.4).
- 3.2 Material. The material shall be a carbon fabric reinforcement impregnated with a carbon filled phenolic resin and supplied as broadgoods. Broadgoods roll widths shall be 36 to 48 inches and shall be rolled onto the paper core with the warp side of the impregnated fabric on the outside face of the rolls (see Figure 1).

3.2.1 Resin. The resin shall conform to the requirements of MIL-R-9299, Grade A, and also to MSFC-SPEC (91LD).

3.2.2 Reinforcement. The reinforcement shall be a carbon fabric conforming to MSFC-SPEC (CCA-3).

3.2.3 Resin Filler. The resin filler shall be carbon powder conforming to MSFC-SPEC (USP-28).

3.3 Properties

3.3.1 Uncured Material. The properties of the uncured material shall be in accordance with Table I.

TABLE I. UNCURED MATERIAL PROPERTIES

PROPERTY	SPECIFICATION LIMITS		RETEST PROPERTIES <u>1/</u>	
	MINIMUM	MAXIMUM	MINIMUM	MAXIMUM
Cloth Content, %	47.0	56.0	----	----
Dry Resin Solids, %	31.0	37.0	----	----
Volatile Content, %	3.5	6.0	3.5	6.5
Carbon Filler Content, %	12.0	18.0	----	----
Resin Flow, %	10.0	20.0	8.0	23.0
Tack, lbs.	5	----	5	---
Sodium Content, ppm	----	750	----	----

1/ Procuring activity testing for extension of material shelf life after six months from date of supplier manufacture.

3.3.2 Cured Material. The properties of the cured material shall be in accordance with Table II.

TABLE II. CURED MATERIAL PHYSICAL AND MECHANICAL PROPERTIES
(AT ROOM TEMPERATURE UNLESS OTHERWISE SPECIFIED)

PROPERTY	LIMITS	
	MINIMUM	MAXIMUM
Density, grams per cubic centimeter (g/cc)	1.400	1.500
Residual Volatiles, percent	0.0	3.0
Resin Content, percent	30.0	38.5
Compressive Strength, psi (edgewise)		
warp direction	40,000	75,000
fill direction	20,000	55,000
Interlaminar Double Shear Strength, psi	3,000	7,500
Thermal Conductivity, Btu/ft-hr-degrees F		
at 250°F with ply	0.10	1.1
across ply	0.10	1.1
Coefficient of Thermal Expansion		
in/in-degrees F x 10 exp -6		
at 400°F across ply	3.0	15.0
with ply	2.0	9.0
Flexural Strength, psi		
warp direction	25,000	55,000
fill direction	20,000	55,000
Tensile Strength, psi		
warp direction	15,000	40,000
fill direction	10,000	35,000

3.4 Age of Material. The material shall be shipped within 3 months of manufacturing date and shall be tested and certified within 60 days of shipment.

3.5 Process of Manufacture. Any change in process or material after initial qualification testing as required by this specification shall be cause to require requalification.

- 3.6 Storage Life. The material shall have a storage life of six months after date of manufacture when stored at 50°F maximum in sealed plastic bags with a 1-unit minimum desiccant bag conforming to MIL-D-3464. After date of receipt of the broadgoods, the maximum cumulative out-of-storage time shall be 30 days at 85°F maximum. Storage life requirements shall apply until the material has been completely cured.
- 3.6.1 Storage Life Extension. For the convenience of the procuring activity, the storage life of broadgoods may be extended for three additional 6-month periods provided the material, upon retest (see 6.5), conforms to the retest requirement for resin flow, volatile content, and tack in Table I. The maximum allowable cumulative storage life of broadgoods shall not exceed 24 months from the date of manufacture of the original broadgoods lot. Each individual roll of broadgoods must be tested for storage life extension.
- 3.6.2 Conditioning for Storage Life Extension Retest. Material which fails to meet the allowable volatile, tack and resin flow retest requirements may be conditioned to extend the storage life by subjecting the rolls which failed to meet the requirements to the following:
- Place broadgoods material in a vacuum chamber supported through core(s) to prevent material damage.
 - Expose material to a vacuum of less than one inch of mercury (Hg) absolute and a temperature of 60-90°F for a period of 7-9 days.
 - After completion of the drying process, obtain samples from outside and inside of each roll and retest for volatile content resin flow and tack.
- Failure of any of the conditioned rolls to meet the retest requirements of Table I shall require the roll to be scrapped.
- 3.6.3 Out-of-Storage Time Extension - The 30-day out-of-storage time at 85°F maximum may be extended for one additional 30-day period provided that each roll and the average of three tests has met the retest requirements for volatile content, resin flow and tack specified in Table I.
- 3.6.4 Preparation and Storage of Samples Selected for Retest. Each lot of samples shall be promptly put into an individual plastic bag, sealed, properly labeled, and stored with the broadgoods it represents. Prior to testing and removal from the plastic bag, the samples shall be conditioned at 60-80°F for two hours minimum. The samples shall be tested within 72 hours after removal from plastic bags and within 14 days after removal from the broadgoods rolls.

3.7 Workmanship

- 3.7.1 Broadgoods. The material shall have no holes, tears, non-uniform weave, resin build-up, contaminants, or other defects which would render more than 5 percent of any roll unsuitable for the intended use.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test results conform to prescribed requirements.
- 4.2 Classification of Inspections. The inspection requirements specified herein are classified as follows:
- a. Qualification inspection (see 4.3)
 - b. Quality conformance inspection (see 4.4).
- 4.3 Qualification Inspection. Three rolls of broadgoods from the first production lot (see 4.4.1.1) shall be subjected to all examinations and tests specified in Table III. The lots subjected to qualification inspection shall be representative of the manufacturing production from the proposed production facility.

TABLE III. QUALIFICATION INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5

TABLE III. QUALIFICATION INSPECTION (CONT'D.)

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Residual Volatiles	3.3.2	4.6.3.3
Resin Content	3.3.2	4.6.3.4
Compressive Strength	3.3.2	4.6.3.5
Interlaminar Shear	3.3.2	4.6.3.6
Thermal Conductivity	3.3.2	4.6.3.7
Coefficient of Thermal Expansion	3.3.2	4.6.3.8
Flexural Strength	3.3.2	4.6.3.9
Tensile Strength	3.3.2	4.6.3.10

4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of all the examinations and tests specified in Table IV performed on each lot.

TABLE IV. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TEST	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Resin Content	3.3.2	4.6.3.4
Compressive Strength (warp direction only)	3.3.2	4.6.3.5

4.4.1 Sampling. Each lot of carbon cloth phenolic shall be sampled for inspection as follows:

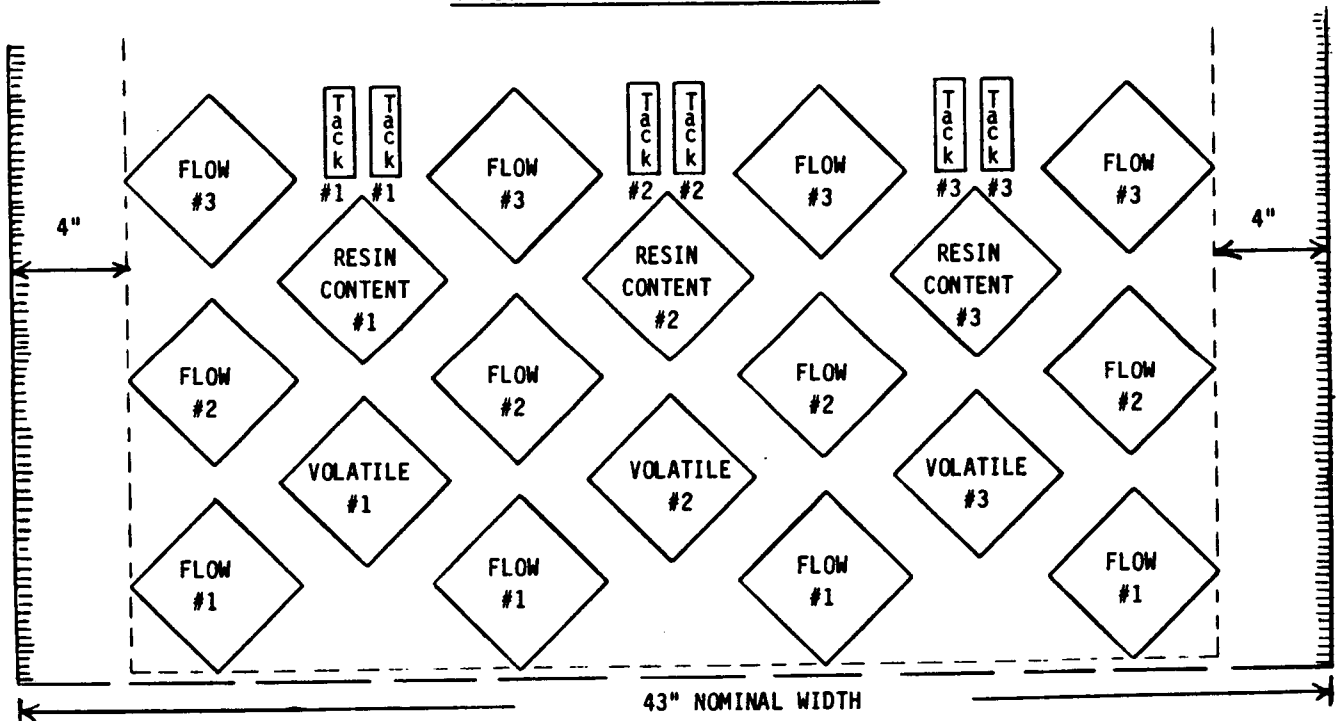
4.4.1.1 Lot. A manufacturing lot of preimpregnated material shall consist of material which is impregnated in one continuous production run, using one batch of the impregnator's resin and additives mixed in one vessel at one time.

4.4.1.1.1 Lot Size. A lot of carbon cloth phenolic shall consist of not more than 15,000 pounds.

4.4.1.2 Lot Acceptance Test Samples.

4.4.1.2.1 Sampling for Uncured Material Properties. Each sample shall be of sufficient size to perform all of the uncured material inspections and tests. Each sample shall be marked with the roll number, lot number, and the location in the roll from which the sample was taken. Each broadgoods roll in the lot shall be sampled at the exposed end of the roll, as shipped.

TYPICAL UNCURED MATERIAL SAMPLING



- 4.4.1.2.2 Sampling for Cured Material Properties. A sample of sufficient size to perform all the cured material inspections and tests specified herein shall be taken from the exposed end of each broadgoods roll selected at random from each lot. The number of rolls to be selected shall be determined as follows:

NUMBER OF ROLLS IN LOT	NUMBER OR ROLLS TO BE SELECTED FOR SAMPLING
1 to 2	All
3 to 40	3
41 to 65	4
66 to 110	5

Failure of a roll sample to conform to the requirements of this specification shall be cause to withdraw that roll and shall require that all other rolls in that lot be tested for only those material properties which failed to meet specification requirements.

- 4.5 Test Requirements. Three tests shall be performed for each of the material properties specified herein on the samples taken from each broadgoods roll sampled. The average values of the test results and at least two of the three individual test values shall be used to determine conformance to the requirements of this specification.

- 4.5.1 Test Conditions. Unless otherwise specified, all tests shall be performed under ambient conditions.

- 4.5.1.1 Supplier Retest. When the average test value of any material property fails to conform to Tables I and II and the cause of the failure can be attributed to improper preparation of the specimen or testing machine anomaly, new tests shall be conducted on a sample from the same broadgoods roll from which the failed specimens were removed.

- 4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20 percent shall be used.

4.6.1 Visual Examination. Each roll of preimpregnated cloth shall be visually examined for conformance to 3.7 and Section 5 before supplier shipment.

4.6.1.1 Visual Examination for Conformance to Special Shipping and Marking Requirements. Temperature record charts of the ambient temperature to which the material was exposed during shipment shall be examined for conformance to 5.2. Marking shall be visually inspected for conformance to 5.3.

4.6.2 Uncured Material Tests.

4.6.2.1 Volatile Content. The volatile content of each sample of the uncured material shall be determined in accordance with the following:

- a. Cut a 16 ± 2 square inch specimen.
- b. Weigh the specimen to the nearest 0.01 gram (g) (W1).
- c. Place specimen in a recirculating oven preheated and stabilized to $325 \pm 10^\circ\text{F}$ for 10 ± 1 minutes. Specimens should be placed to get adequate air circulation.
- d. Remove the specimen and within one (1) minute, weigh to the nearest 0.01 g (W2).
- e. Calculate percent volatiles as follows:

$$\text{Percent volatiles} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g

W2 = final weight of specimen, g

4.6.2.2 Resin Solids, Filler Content, and Cloth Content. Resin solids, shall be determined for each sample in accordance with the soxhlet extraction method specified in ASTM C 613 and the following:

- a. Precondition a paper thimble at $325 \pm 5^\circ\text{F}$ for 120 minutes. Weigh immediately to .001 gm (W1) and store in a desiccator with the recorded weight until ready to use.
- b. Reweigh the thimble immediately before addition of the sample (W2) so an accurate sample weight is established. Place a bias cut 2.0 x 4.0 inch prepreg sample into the thimble and weigh to .001 gm (W3).

- c. Using the volatile content obtained in 4.6.2.1e, calculate the devolatilized sample weight by using the formula:

$$W = \text{devolatilized sample weight} = (W3 - W2) \times \left(1 - \frac{V}{100}\right)$$

Where W2 = Thimble weight immediately before sample addition, gm.

W3 = Thimble weight plus sample, gm.

V = Volatile content, wt. %

- d. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, allow the solvent to drain, and dry to constant weight at $325 \pm 5^\circ\text{F}$ for 2 hours minimum in a forced air oven. Remove from the oven in sets of three, and immediately weigh to .001 gm (W4).
- e. If filler has appeared in the extract, determine the filler weight lost by the filtering and crucible technique in ASTM C-613, and add the recovered filler weight to the dried thimble plus sample weight (W4).
- f. Calculate the dry resin content (resin solids) as follows:

$$\text{DRC, wt. \%} = 100 \left(1 - \frac{W4 - W1}{W}\right)$$

Where W = Devolatilized sample weight, gm.

W1 = Weight of dried thimble, gm.

W4 = Weight of thimble plus extracted sample, gm.

- g. Filler content can be calculated by utilizing the filler content in the resin mix before impregnation as follows:

$$\text{Filler content, wt. \%} = \frac{(\text{DRC}) (\text{FC})}{(100 - \text{FC})}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content of the resin mix

- h. Cloth content can be calculated as follows:

$$\text{Cloth content, wt. \%} = 100 - \text{DRC} - \text{FC}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content 4.6.2.2g

4.6.2.2.1 As an alternative method for determining filler content and cloth content, the following procedure may be used:

- a. Follow the procedures of 4.6.2.2 thru 4.6.2.2c.
- b. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, and allow the solvent to drain.
- c. Carefully open the thimble, remove the cloth and place the cloth in the bottom of a clean 250 ml beaker. Add 20-25 ml of ethyl alcohol and gently stir to remove any remaining filler.
- d. Transfer the liquid solution back to the original thimble, removing any visible fibers and keeping the fibers with the fabric.
- e. Rinse the fabric with ethyl alcohol several times to ensure complete removal of the filler from the fabric and add the rinses back to the original thimble. Make the final rinse acetone to remove all traces of ethyl alcohol.
- f. Dry the fabric in an oven for a minimum of 30 minutes at 325°F, cool in a desiccator to room temperature and reweigh to the nearest .001 gm (W5). This is the dry cloth weight.
- g. Close the top of the thimble and dry to a constant weight at 325 ± 5°F for 2 hours minimum in a forced air oven. Remove from the oven and immediately weigh to .001 gm (W6).
- h. Calculate the resin solids, filler content and cloth content as follows:

$$\text{Filler content, wt. \%} = \left(\frac{W6 - W1}{W} \right) \times 100$$

$$\text{Cloth content, wt. \%} = \left(\frac{W5}{W} \right) \times 100$$

$$\text{Resin solids content, wt. \%} = 100 - \left(\frac{W6 + W5 - W1}{W} \right)$$

Where W = devolatilized sample weight from 4.6.2.2

W1 = weight of dried thimble, gm.

W5 = weight of dried cloth, gm.

W6 = weight of thimble plus filler, gm.

4.6.2.3 Resin Flow. Resin flow of each sample shall be determined in accordance with the following:

- a. Cut four 4-inch by 4-inch \pm 1/8-inch squares across the width of the fabric. All squares shall be bias cut to eliminate fiber loss in testing. Stack the squares uniformly on each other to make a specimen.
- b. Weigh the specimen of four plies to the nearest 0.01 g and record as W1.
- c. Place the specimen between release film. Preheat the press to $325 \pm 10^\circ\text{F}$, position the specimen in the middle of the press plate and apply the press load of 150 ± 10 pounds per square inch gauge (psig) immediately. Press-load the specimen for a minimum of 10 minutes at $325 \pm 10^\circ\text{F}$.
- d. Remove the specimen from the press and cool to ambient temperature.
- e. Using the edge of a metal plate or knife, scrape off the resin flash to the original size of the specimen. Do not remove any reinforcement from the original dimensions.

NOTE: Include any fibers which may be displaced and scraped off during the process in the weighed back specimen weight (W2).

- f. Reweigh the specimen to the nearest 0.01 g and record as W2.
- g. Calculate the percent resin flow as follows:

$$\text{Percent flow} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g
W2 = final weight of specimen, g

4.6.2.4 Tack. Tack shall be determined in accordance with the following:

- a. Cut two 1.0 x 3" strips and place them lengthwise with a 1.0" overlap (wrap to fill side) into a sheet of folded cellophane.
- b. Place the wrapped specimen onto a preheated 180°F hot plate and cover the overlapped area with a 2" x 2" x $\frac{1}{4}$ " metal block (weighing $125 \text{ gms.} \pm 5 \text{ gms.}$).
- c. Allow the specimen to heat for 30 ± 2 seconds.

- d. Remove the 125-gram block and immediately (within 5 seconds) place the specimen, with the cellophane intact, into a room temperature press and apply 50 ± 2 lbs. pressure to the 1" overlapped area for approximately $1\frac{1}{2}$ minutes, while the specimen cools. The press platens shall be covered with insulating non-metallic laminates to allow controlled cooling.
- e. Using an appropriate tensile testing device, apply a constant rate of loading to obtain sample failure within 5 to 10 seconds. Record and report the average load at failure of three determinations.

4.6.2.5 Sodium Content. Sodium content shall be determined in accordance with the following:

NOTE: Care should be exercised not to contaminate the sample with sodium. Samples should not be touched with bare hands.

- a. Take specimens which weigh approximately 3 g. Place test specimens in individual beakers and dry in an air circulating oven at $225 \pm 5^\circ\text{F}$ for a minimum of one hour.
- b. Remove the specimens from the oven and cool in a desiccator.
- c. Weigh approximately 2 g of each cooled specimen to the nearest 0.1 milligram (mg) into separate tared ceramic or platinum crucibles.
- d. Heat the crucibles in a muffle furnace at $600 \pm 15^\circ\text{C}$ for 16-18 hours or until constant weight is achieved.
- e. Remove the crucibles containing the ashed specimens, place in a desiccator and cool.
- f. Wash down the walls of each crucible with distilled water and add approximately 5 ml concentrated hydrochloric acid to each.
- g. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a 1 liter volumetric flask and dilute to volume with distilled water.
- h. Determine the sodium content of each flask by flame emission at 589 nanometers (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

4.6.3 Cured Material Tests.

4.6.3.1 Preparation of Test Panels. Cured material tests shall be determined on test panels prepared in accordance with the following, when referenced:

- a. Cut sufficient plies to make the specified panel thickness and minimum size required, maintaining and marking uniform orientation of cloth fiber direction.
- b. Wrap the stack of plies $1\frac{1}{2}$ times with a suitable release film such that the upper and lower surfaces and two sides are completely encased by the release film. The open ends allow for degassing and resin flow.
- c. Center the wrapped plies between two $1/8$ " thick smooth steel caul plates and center the caul plates in a press preheated to $325 \pm 10^\circ\text{F}$, starting the cure timer.
- d. Apply contact pressure (nominal 50-lb. load) and maintain for $30 \pm$ seconds. Increase the load rapidly to approximately 200-lb. load and immediately release the pressure until a clearance can be observed above the top caul plate.
- e. Immediately reapply contact pressure and "bump" the laminate as indicated above for one additional cycle.
- f. Increase the pressure slowly (over approximately 4 minutes) to 1000 ± 50 psi, controlling the rate of pressure application to minimize resin flow at the laminate edge.
- g. Maintain the pressure and temperature for 120 ± 15 minutes. Turn-off the platen heaters and allow the laminate to cool to below 150°F under pressure. Remove from the press, remove release film and resin flash. Do not use the outer $\frac{1}{2}$ " of the test panel for preparation of specimens, unless specifically directed.

4.6.3.2 Density. Density shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches by 5 inches minimum, prepared as per 4.6.3.1.
- b. Cut a specimen 1-inch by 1-inch by panel thickness at random from the test panel. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry 20 minutes, minimum, before testing.
- c. Weigh and record the weight of the specimen in air to the nearest 0.1 mg (W1).

d. Weigh the specimen in water at $73 \pm 3^\circ\text{F}$ and record the weight to the nearest 0.1 mg using a wire and sinker if required (W2).

e. Calculate density using water as follows:

$$\text{Density (g/cc)} = \frac{0.9975 W1}{(W1 + W3 - W2)}$$

Where: W1 = weight of specimen in air

W2 = weight of specimen, wire, and sinker suspended in water

W3 = weight of wire and sinker suspended in water to the same depth as in the W2 determination to the nearest 0.1 mg.

4.6.3.3 Residual Volatile Content. Residual volatile content shall be determined in accordance with the following:

a. Cut sufficient plies to produce a test panel 0.250 ± 0.050 inches thick and 4 inches by 4 inches, prepared as per 4.6.3.1.

b. Cut a specimen 1.0 inch by 1.0 inch by panel thickness from the test panel; do not machine the top and bottom molded surfaces. Wipe the specimen clean using MEK and allow to air dry 20 minutes minimum.

c. Place the specimen in a desiccator and desiccate for 18 hours minimum.

d. Weigh the specimen to the nearest 0.01 gram and record as W1.

e. Place the specimen in an air circulating oven, preheated and stabilized at $325 \pm 10^\circ\text{F}$. Maintain the specimen at a temperature of $325 \pm 10^\circ\text{F}$ for 4 hours.

f. Remove the specimen from the oven and cool in a desiccator for approximately 30 minutes or until the specimen reaches room temperature.

g. Reweigh the specimen to the nearest 0.01 gram. Record as W2.

h. Calculate the percent residual volatiles as follows:

$$\text{Residual volatile, \%} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W_1 = original weight of specimen, g

W_2 = devolatilized weight of specimen, g

4.6.3.4 Resin Content. Resin content shall be determined in accordance with the following:

a. Prepare test specimens as follows:

- (1) Cut 32 rectangles (1/2 inch by 5 inches nominal) from a prepreg sample.
- (2) Stage (see 6.3.1) the 32 rectangles at $255 \pm 5^\circ\text{F}$ for approximately 20 minutes to reduce flow.
- (3) Using the 32 rectangles, mold a bar 1/2 inch by 1/2 inch by 5 inches.
- (4) Cure the specimen at $325 \pm 10^\circ\text{F}$ and 1000 ± 50 psi for 10 ± 1 minutes.

NOTE: If there is any resin flash, repeat and use a longer staging time.

- (5) Cut the bar into approximately three equal lengths to form three test specimens.

b. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry for 20 minutes, minimum, before testing.

c. Record specimen weight to the nearest 0.001 g (W).

d. Place the test specimen in the Vycor test tube and tightly attach the tube to the test apparatus (see Figure 2).

e. Start the vacuum pump and evacuate the system to less than two inches of Hg absolute. Check for air leaks by clamping off the hose to the vacuum pump and noting the manometer. The leak rate must be less than 0.20 inch of Hg per minute. Recheck joints until this level is achieved.

f. With the vacuum on and the pressure stabilized, place the Fisher burner under the specimen. Adjust flame area to completely cover the sample.

NOTE: A tube furnace maintained at $1500 \pm 25^\circ\text{F}$ may be used in place of the Fisher burner.

g. Continue pyrolysis for approximately 30 minutes or until the manometer or gauge reading is the same reading as it was prior to pyrolysis, whichever is longer.

NOTE: If residue collects in the Vycor test tube, place the Fisher burners under those areas until the burnout is complete.

- h. When pyrolysis is complete, turn off burners and cool under vacuum until specimen is at room temperature.
- i. When the specimen is cool, turn off vacuum and carefully let air into the system. When pressure equilibrium is reached, remove specimen from test tube and weigh to the nearest 0.001 g (W1).
- j. Calculate dry resin content as follows:

$$\text{Dry resin content, percent} = \left(1.00 - \frac{W1}{W} \right) \times 100 \times K$$

Where: W1 = weight of specimen after pyrolysis, g

W2 = weight of specimen prior to pyrolysis, g

K = a constant as shown in Table V

TABLE V. K FACTORS

U.S. POLYMERIC FM 5055B
2.008

4.6.3.5 Compressive Strength. Compressive strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches minimum by 5 inches minimum, prepared as per 4.6.3.1.
- b. Machine a specimen to the dimensions shown in Figure 3.
- c. Measure the width and thickness of the specimen to the nearest 0.001 inch in the gauge section area. Calculate and record the cross-sectional area.
- d. Place the specimen in the support jig (see Figure 4), centered so the ends project an even amount beyond both ends of the jig. Tighten finger tight only.

- e. Place the specimen and the support jig in the testing machine and align so that the specimen ends are parallel with the surface of the compression tool. Adjust the crosshead of the testing machine until it just contacts the top of the specimen.
- f. Set the speed control at an indicated crosshead rate of 0.050 inch per minute and start the machine.
- g. Record the maximum load carried by the specimen during the test.
- h. Calculate the compressive strength by dividing the maximum load by the original cross-section area.

4.6.3.6 Interlaminar Shear. Interlaminar shear strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 1.5 ± 0.25 inch thick across the plies by minimum dimensions of 4.0 by 4.0 inches.
- b. Cure at 1000 ± 50 psi and $325 \pm 10^\circ\text{F}$ for 120 ± 15 minutes.
- c. After cure, remove the panel from the press and allow the panel to cool to room temperature.
- d. Cut a specimen of sufficient size to produce a 0.375 inch diameter by 1.125 inch minimum length finished specimen with the length being perpendicular to the plies from the test panel.

NOTE: Do not use the outer 1/2 inch of the test panel for specimen preparation.

- e. Center the specimen to get the length of the cylindrical specimen perpendicular to the plies and machine the specimens to a diameter of $0.375 +0.000, -0.002$ inch diameter.
- f. Measure the diameter of the test specimen and calculate the cross-sectional area (A).
- g. Place the specimen in the 0.375 inch diameter three plate double-shear jig shown in Figure 5.
- h. Place the shear fixture in the testing machine, align, and pin the ends of the fixture.
- i. Set the machine at an indicated crosshead speed of 0.015 to 0.025 inch per minute and test.

- j. Record the maximum load carried by the specimen during the test and record as L.
- k. Calculations:

$$\text{Interlaminar shear (psi)} = \frac{L}{2A}$$

Where: L = the maximum load carried by the specimen during testing in pounds

A = the original cross-sectional area of the specimen in square inches.

4.6.3.7 Thermal Conductivity. Thermal conductivity shall be determined in accordance with ASTM C 177 or equivalent and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Sample thickness is dependent on the dimensions of the heating unit and shall be restricted to limit the theoretical error to 1%.
- c. Determine both cross ply and with ply thermal conductivity at 250°F. Faces of the specimen must be parallel to 1% of specimen thickness.

4.6.3.8 Coefficient of Thermal Expansion. The coefficient of thermal expansion (see 6.3.2) shall be determined in accordance with ASTM E 831 and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Determine the coefficient of thermal expansion in both the with ply or parallel, and cross ply or perpendicular direction of the specimens.

4.6.3.9 Flexural Strength. Flexural strength shall be determined in accordance with ASTM D 790 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each of the desired yarn orientations, prepared as per 4.6.3.1.
- b. Test per method I, procedure A with a span-to-depth ratio of 16 to 1 with three specimens for each sample.

4.6.3.10 Tensile Strength. Tensile strength shall be determined in accordance with ASTM D 638 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each desired yarn orientations, as per 4.6.3.1.
- b. Test with Type I specimens, with dimensions and tolerances for material thickness 0.28 or under, and testing speed of 0.2 in/min.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. The rolls shall be in sealed bags conforming to MIL-B-131, Type 1, Class 1 barrier material. Each broadgoods roll shall be separately wrapped with a 1-unit, minimum, dessicant bag conforming to MIL-D-3464 enclosed within each bag.

5.1.1 Broadgoods Shipping Container. The broadgoods shall be shipped in a container that can be opened from the top and will suspend both ends of the roll in the container.

5.2 Special Shipping Requirements. During shipment, the material temperature shall be maintained at or below 90°F. The ambient temperature to which the material is exposed during shipment shall be recorded with a temperature recording device provided by the procuring activity and placed with each lot shipment by the supplier.

5.3 Marking. Unless otherwise specified in the purchase document, each container and roll shall be marked for identification and shipment and shall include the following:

- a. Supplier lot number and material name
- b. Purchase document number
- c. Acceptance stock and lot number
- d. Number of rolls
- e. Roll net weight
- f. Container identification number
- g. Date of manufacture
- h. Mark each roll on inside of spool with supplier name, roll number, lot number and material name with ink stamp, stencil or adhesive label.

- i. This specification number and revision letter
- j. Mark each container on two opposite sides with the following:

**TEMPERATURE SENSITIVE MATERIAL
DO NOT EXPOSE TO TEMPERATURES EXCEEDING 90°F**

6. NOTES

- 6.1 Intended Use. The carbon cloth phenolic covered by this specification is intended for use in the manufacture of rocket motor nozzle components.
- 6.2 Ordering Data. Purchase documents should specify the following:
 - a. Title, number, and date of this specification
 - b. Special marking, packing, packaging, and shipping
 - c. Data retention requirements
 - d. Broadgoods roll width
 - e. Qualification sampling requirements (if required)
 - f. Number of copies of inspection and test data
 - g. Type of material: broadgoods or trimmed broadgoods
 - h. Acceptance stock and lot number
- 6.3 Definitions.
 - 6.3.1 Stage. A method of stabilizing and retaining the phenolic polymer in a solid state on the carbon cloth by temperature cycling.
 - 6.3.2 Coefficient of Thermal Expansion. The slope of the straight line intersecting the linear expansion curve at 75°F and 400°F expressed as change in length per unit length per °F.
- 6.4 Qualification. The procuring activity reserves the right to purchase only those products which have been previously tested and found to meet the requirements of this specification and which have proved satisfactory in one full scale nozzle static test. Prospective suppliers whose products have not been tested and found satisfactory are required to have their products tested in order that they may be considered eligible to be awarded contracts for the product covered by this specification.
- 6.5 Storage. After receipt of the material, the procuring activity is responsible for storage and for retesting to determine storage life extension.

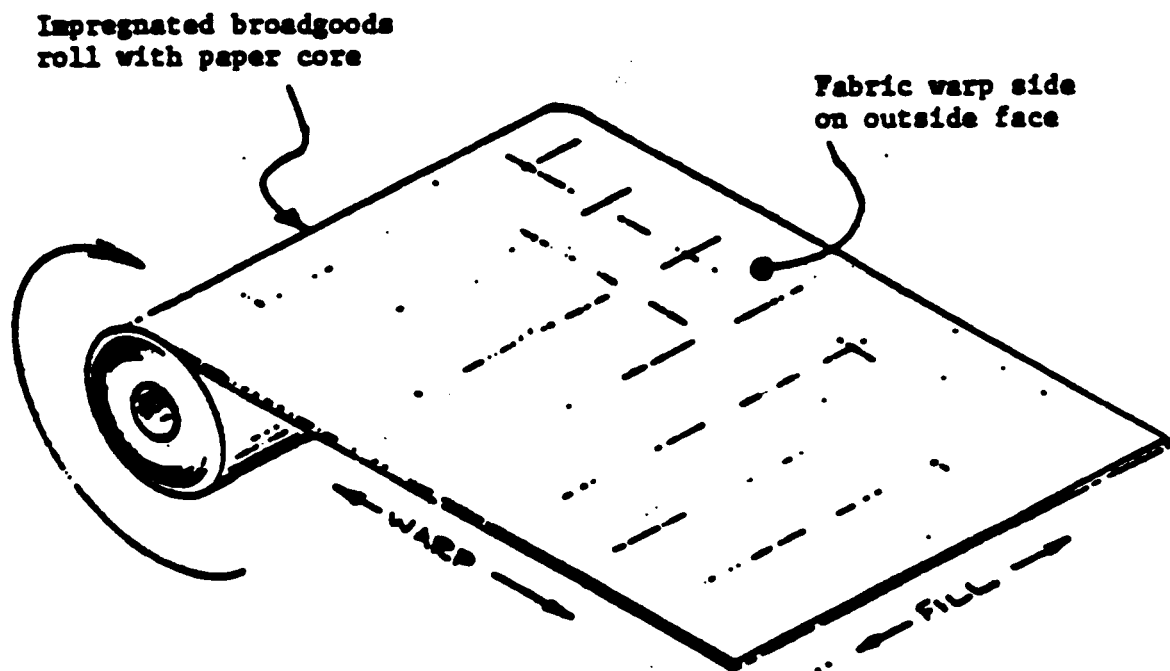


Figure 1. Requirements for Broadgoods Rolls

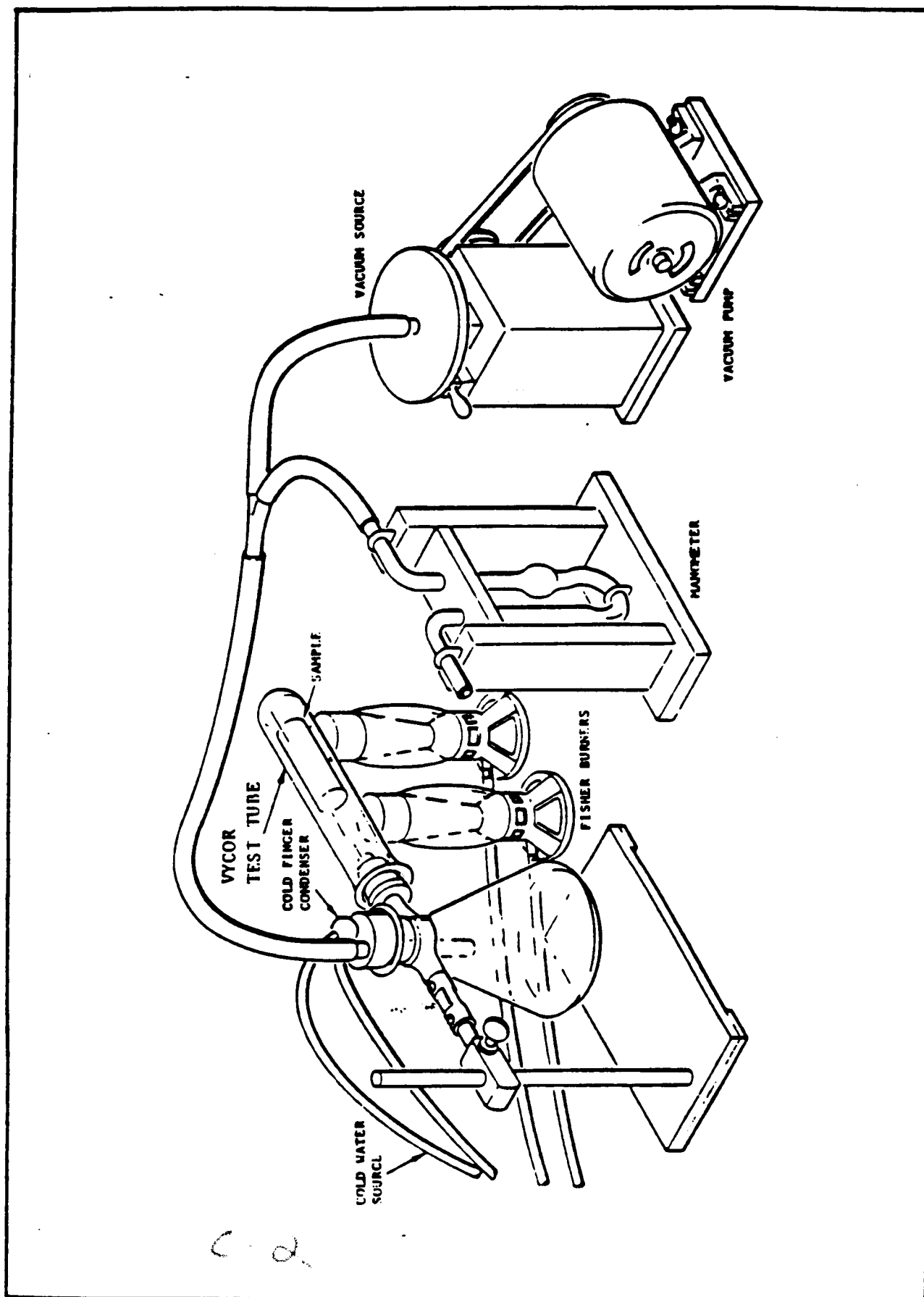
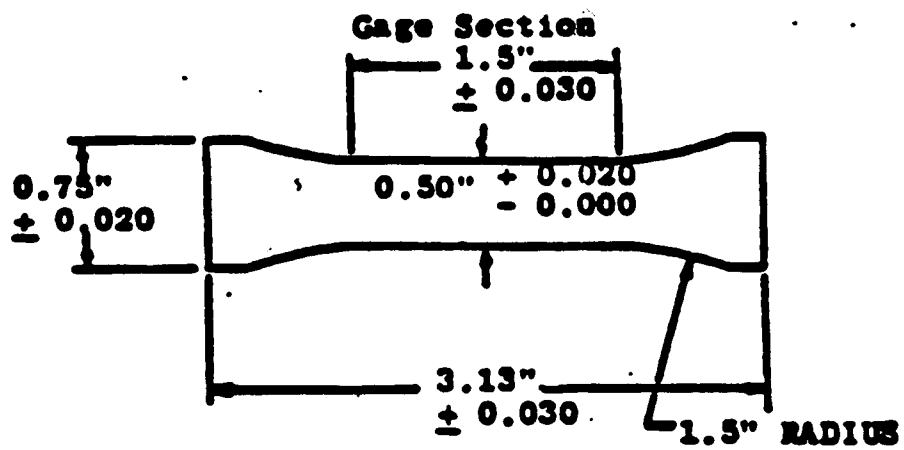


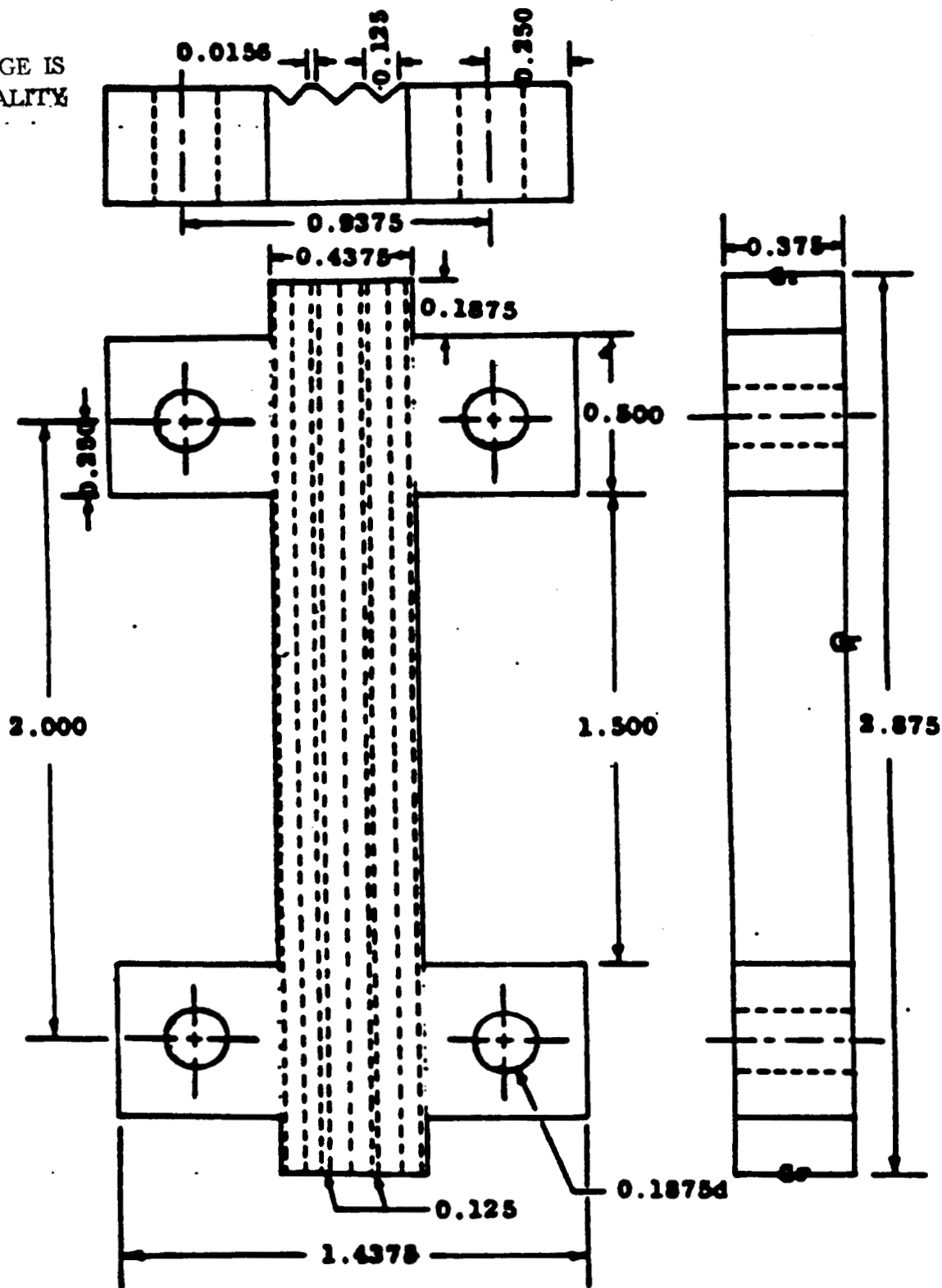
Figure 2. Resin Content Apparatus Setup



Test specimen thickness = 0.125 ± 0.025 inch

Figure 3. Compressive Strength Test Specimen

ORIGINAL PAGE IS
OF POOR QUALITY



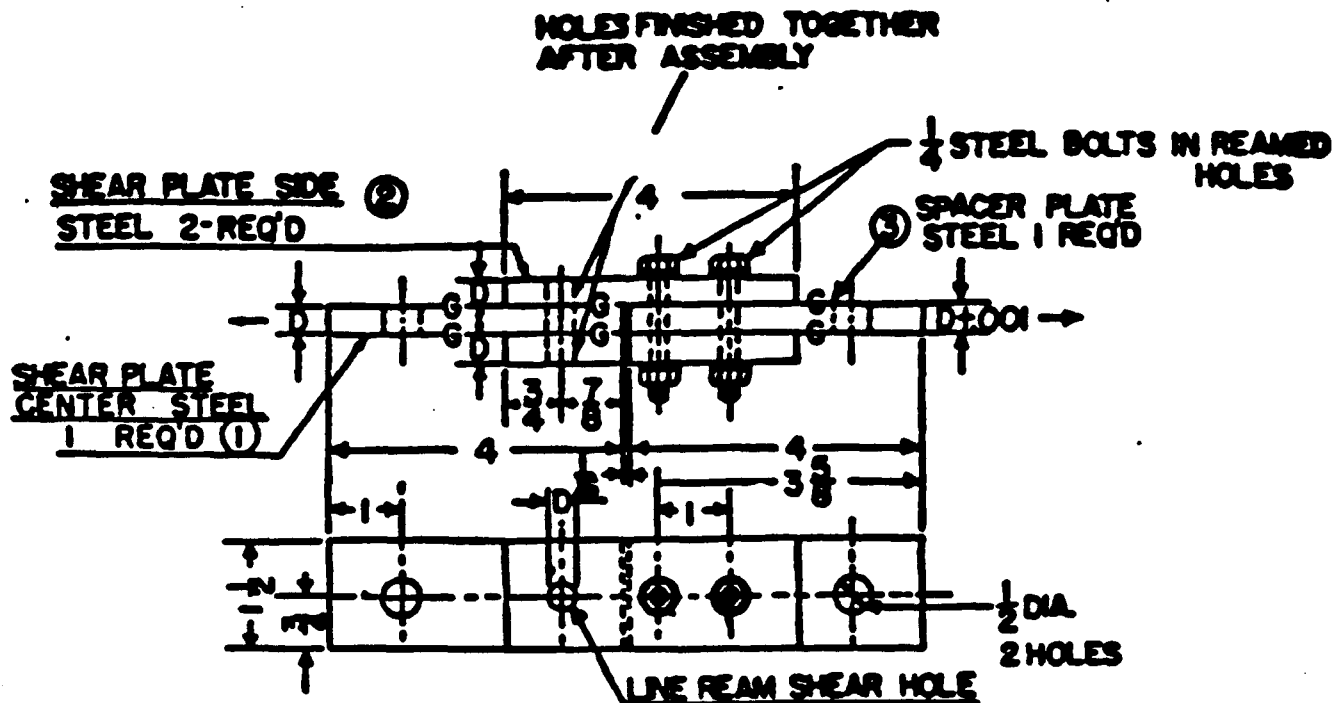
NOTE 1. - Cold rolled steel

NOTE 2. - Furnished four steel machine screws and nuts, round head, slotted, length 1-1/4 in.

NOTE 3. - Grind surfaces denoted "Gr"

Figure 4. Support Jig Details

ORIGINAL PAGE IS
OF POOR QUALITY



- NOTES:
1. D is nominal finish of test cylinder.
 2. Finish: machine after heat treatment.
 3. Grind surfaces denoted G.
 4. Spacer plate (3) to be 0.001 inch thicker than (1).
 5. Edges of shear hole must be sharp.
 6. All dimensions in inches; tolerances—fractions $\pm 1/64$; specimen diameter = $D + 0.000, -0.001$; diameter shear hole = $D - 0.000, -0.001$.
 7. Alignment of shear holes must be maintained.
 8. Material: steel, heat-treated to Rockwell C 35-40.

Figure 5. Double Shear Jig

1 February 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

SPUN PAN PHENOLIC CLOTH, 8.5 oz/yd², PREIMPREGNATED

1. SCOPE

- 1.1 Scope. This specification covers one type of carbon cloth reinforcement preimpregnated with a carbon filled phenolic resin.

2. APPLICABLE DOCUMENTS

- 2.1 Government Documents. The following documents of the issue in effect on date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

SPECIFICATIONS

Military

MIL-B-131	Barrier Materials, Watervapor-proof, Grease-proof, Flexible, Heat-sealable
MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification
MIL-R-9299	Resin, Phenolic, Laminating

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

- 2.2 Non-Government Documents. Unless otherwise specified, the following documents of the latest approved issues form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (USP-28) Filler, Carbon Black

MSFC-SPEC (SWB-8) Reinforcement, Carbon, Spun PAN, 8.5 oz/yd²

MSFC-SPEC (91LD) Resin, Phenolic

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812.)

American Society for Testing and Materials (ASTM)

ASTM C 177	Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate, Standard Test Method for
ASTM C 613	Resin Content of Carbon and Graphite Ppregs by Solvent Extraction, Standard Test Method for
ASTM D 638	Tensile Properties of Plastics, Standard Test Method for
ASTM D 790	Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials, Standard Test Methods for
ASTM E 831	Linear Thermal Expansion of Solid Materials by Thermodilatometry, Standard Test Method for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

- 3.1 Qualification. The material furnished under this specification shall be a product which has been tested and has passed the qualification inspection specified herein (see 4.3 and 6.4).
- 3.2 Material. The material shall be a carbon fabric reinforcement impregnated with a carbon filled phenolic resin and supplied as broadgoods. Broadgoods roll widths shall be 36 to 48 inches and shall be rolled onto the paper core with the warp side of the impregnated fabric on the outside face of the rolls (see Figure 1).

3.2.1 Resin. The resin shall conform to the requirements of MIL-R-9299, Grade A, and also to MSFC-SPEC (91LD).

3.2.2 Reinforcement. The reinforcement shall be a carbon fabric conforming to MSFC-SPEC (SWB-8).

3.2.3 Resin Filler. The resin filler shall be carbon powder conforming to MSFC-SPEC (USP-28).

3.3 Properties

3.3.1 Uncured Material. The properties of the uncured material shall be in accordance with Table I.

TABLE I. UNCURED MATERIAL PROPERTIES

PROPERTY	SPECIFICATION LIMITS		RETEST	PROPERTIES <u>1/</u>
	MINIMUM	MAXIMUM	MINIMUM	MAXIMUM
Cloth Content, %	39.0	53.0	----	----
Dry Resin Solids, %	33.0	43.0	----	----
Volatile Content, %	3.5	6.0	3.5	7.0
Carbon Filler Content, %	11.0	19.0	----	----
Resin Flow, %	8.0	20.0	8.0	23.0
Tack, lbs.	5	----	5	---
Sodium Content, ppm	----	100	----	----

1/ Procuring activity testing for extension of material shelf life after six months from date of supplier manufacture.

3.3.2 Cured Material. The properties of the cured material shall be in accordance with Table II.

TABLE II. CURED MATERIAL PHYSICAL AND MECHANICAL PROPERTIES
(AT ROOM TEMPERATURE UNLESS OTHERWISE SPECIFIED)

PROPERTY	LIMITS	
	MINIMUM	MAXIMUM
Density, grams per cubic centimeter (g/cc)	1.440	1.600
Residual Volatiles, percent	0.0	3.0
Resin Content, percent	32.0	44.0
Compressive Strength, psi (edgewise)		
warp direction	20,000	45,000
fill direction	TBD	TBD
Interlaminar Double Shear Strength, psi	2,000	5,500
Thermal Conductivity, Btu/ft-hr-degrees F		
at 250°F with ply	TBD	TBD
across ply	TBD	TBD
Coefficient of Thermal Expansion		
in/in-degrees F x 10 exp -6		
at 400°F across ply	5.0	20.0
with ply	-5.0	5.0
Flexural Strength, psi		
warp direction	30,000	60,000
fill direction	TBD	TBD
Tensile Strength, psi		
warp direction	15,000	45,000
fill direction	TBD	TBD

3.4 Age of Material. The material shall be shipped within 3 months of manufacturing date and shall be tested and certified within 60 days of shipment.

3.5 Process of Manufacture. Any change in process or material after initial qualification testing as required by this specification shall be cause to require requalification.

- 3.6 Storage Life. The material shall have a storage life of six months after date of manufacture when stored at 50°F maximum in sealed plastic bags with a 1-unit minimum desiccant bag conforming to MIL-D-3464. After date of receipt of the broadgoods, the maximum cumulative out-of-storage time shall be 30 days at 85°F maximum. Storage life requirements shall apply until the material has been completely cured.
- 3.6.1 Storage Life Extension. For the convenience of the procuring activity, the storage life of broadgoods may be extended for three additional 6-month periods provided the material, upon retest (see 6.5), conforms to the retest requirement for resin flow, volatile content, and tack in Table I. The maximum allowable cumulative storage life of broadgoods shall not exceed 24 months from the date of manufacture of the original broadgoods lot. Each individual roll of broadgoods must be tested for storage life extension.
- 3.6.2 Conditioning for Storage Life Extension Retest. Material which fails to meet the allowable volatile, tack and resin flow retest requirements may be conditioned to extend the storage life by subjecting the rolls which failed to meet the requirements to the following:
- a. Place broadgoods material in a vacuum chamber supported through core(s) to prevent material damage.
 - b. Expose material to a vacuum of less than one inch of mercury (Hg) absolute and a temperature of 60-90°F for a period of 7-9 days.
 - c. After completion of the drying process, obtain samples from outside and inside of each roll and retest for volatile content resin flow and tack.
- Failure of any of the conditioned rolls to meet the retest requirements of Table I shall require the roll to be scrapped.
- 3.6.3 Out-of-Storage Time Extension - The 30-day out-of-storage time at 85°F maximum may be extended for one additional 30-day period provided that each roll and the average of three tests has met the retest requirements for volatile content, resin flow and tack specified in Table I.
- 3.6.4 Preparation and Storage of Samples Selected for Retest. Each lot of samples shall be promptly put into an individual plastic bag, sealed, properly labeled, and stored with the broadgoods it represents. Prior to testing and removal from the plastic bag, the samples shall be conditioned at 60-80°F for two hours minimum. The samples shall be tested within 72 hours after removal from plastic bags and within 14 days after removal from the broadgoods rolls.

3.7 Workmanship

3.7.1 Broadgoods. The material shall have no holes, tears, non-uniform weave, resin build-up, contaminants, or other defects which would render more than 5 percent of any roll unsuitable for the intended use.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test results conform to prescribed requirements.

4.2 Classification of Inspections. The inspection requirements specified herein are classified as follows:

- a. Qualification inspection (see 4.3)
- b. Quality conformance inspection (see 4.4).

4.3 Qualification Inspection. Three rolls of broadgoods from the first production lot (see 4.4.1.1) shall be subjected to all examinations and tests specified in Table III. The lots subjected to qualification inspection shall be representative of the manufacturing production from the proposed production facility.

TABLE III. QUALIFICATION INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5

TABLE III. QUALIFICATION INSPECTION (CONT'D.)

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Residual Volatiles	3.3.2	4.6.3.3
Resin Content	3.3.2	4.6.3.4
Compressive Strength	3.3.2	4.6.3.5
Interlaminar Shear	3.3.2	4.6.3.6
Thermal Conductivity	3.3.2	4.6.3.7
Coefficient of Thermal Expansion	3.3.2	4.6.3.8
Flexural Strength	3.3.2	4.6.3.9
Tensile Strength	3.3.2	4.6.3.10

4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of all the examinations and tests specified in Table IV performed on each lot.

TABLE IV. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TEST	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Resin Content	3.3.2	4.6.3.4
Compressive Strength (warp direction only)	3.3.2	4.6.3.5

4.4.1 Sampling. Each lot of carbon cloth phenolic shall be sampled for inspection as follows:

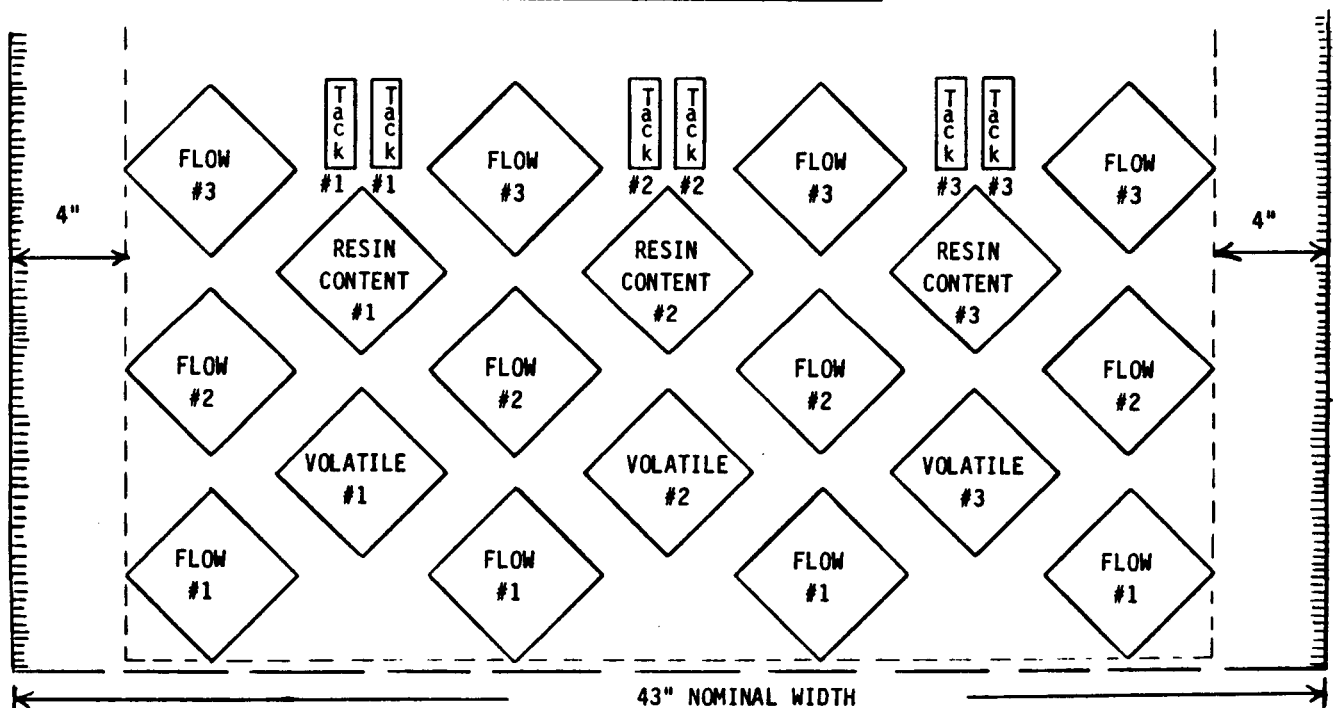
4.4.1.1 Lot. A manufacturing lot of preimpregnated material shall consist of material which is impregnated in one continuous production run, using one batch of the impregnator's resin and additives mixed in one vessel at one time.

4.4.1.1.1 Lot Size. A lot of carbon cloth phenolic shall consist of not more than 15,000 pounds.

4.4.1.2 Lot Acceptance Test Samples.

4.4.1.2.1 Sampling for Uncured Material Properties. Each sample shall be of sufficient size to perform all of the uncured material inspections and tests. Each sample shall be marked with the roll number, lot number, and the location in the roll from which the sample was taken. Each broadgoods roll in the lot shall be sampled at the exposed end of the roll, as shipped.

TYPICAL UNCURED MATERIAL SAMPLING



- 4.4.1.2.2 Sampling for Cured Material Properties. A sample of sufficient size to perform all the cured material inspections and tests specified herein shall be taken from the exposed end of each broadgoods roll selected at random from each lot. The number of rolls to be selected shall be determined as follows:

NUMBER OF ROLLS IN LOT	NUMBER OR ROLLS TO BE SELECTED FOR SAMPLING
1 to 2	All
3 to 40	3
41 to 65	4
66 to 110	5

Failure of a roll sample to conform to the requirements of this specification shall be cause to withdraw that roll and shall require that all other rolls in that lot be tested for only those material properties which failed to meet specification requirements.

- 4.5 Test Requirements. Three tests shall be performed for each of the material properties specified herein on the samples taken from each broadgoods roll sampled. The average values of the test results and at least two of the three individual test values shall be used to determine conformance to the requirements of this specification.

- 4.5.1 Test Conditions. Unless otherwise specified, all tests shall be performed under ambient conditions.

- 4.5.1.1 Supplier Retest. When the average test value of any material property fails to conform to Tables I and II and the cause of the failure can be attributed to improper preparation of the specimen or testing machine anomaly, new tests shall be conducted on a sample from the same broadgoods roll from which the failed specimens were removed.

- 4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20 percent shall be used.

4.6.1 Visual Examination. Each roll of preimpregnated cloth shall be visually examined for conformance to 3.7 and Section 5 before supplier shipment.

4.6.1.1 Visual Examination for Conformance to Special Shipping and Marking Requirements. Temperature record charts of the ambient temperature to which the material was exposed during shipment shall be examined for conformance to 5.2. Marking shall be visually inspected for conformance to 5.3.

4.6.2 Uncured Material Tests.

4.6.2.1 Volatile Content. The volatile content of each sample of the uncured material shall be determined in accordance with the following:

- a. Cut a 16 ± 2 square inch specimen.
- b. Weigh the specimen to the nearest 0.01 gram (g) (W1).
- c. Place specimen in a recirculating oven preheated and stabilized to $325 \pm 10^\circ\text{F}$ for 10 ± 1 minutes. Specimens should be placed to get adequate air circulation.
- d. Remove the specimen and within one (1) minute, weigh to the nearest 0.01 g (W2).
- e. Calculate percent volatiles as follows:

$$\text{Percent volatiles} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g

W2 = final weight of specimen, g

4.6.2.2 Resin Solids, Filler Content, and Cloth Content. Resin solids, shall be determined for each sample in accordance with the soxhlet extraction method specified in ASTM C 613 and the following:

- a. Precondition a paper thimble at $325 \pm 5^\circ\text{F}$ for 120 minutes. Weigh immediately to .001 gm (W1) and store in a desiccator with the recorded weight until ready to use.
- b. Reweigh the thimble immediately before addition of the sample (W2) so an accurate sample weight is established. Place a bias cut 2.0×4.0 inch prepreg sample into the thimble and weigh to .001 gm (W3).

- c. Using the volatile content obtained in 4.6.2.1e, calculate the devolatilized sample weight by using the formula:

$$W = \text{devolatilized sample weight} = (W3 - W2) \times \left(1 - \frac{V}{100}\right)$$

Where W2 = Thimble weight immediately before sample addition, gm.

W3 = Thimble weight plus sample, gm.

V = Volatile content, wt. %

- d. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, allow the solvent to drain, and dry to constant weight at $325 \pm 5^\circ\text{F}$ for 2 hours minimum in a forced air oven. Remove from the oven in sets of three, and immediately weigh to .001 gm (W4).
- e. If filler has appeared in the extract, determine the filler weight lost by the filtering and crucible technique in ASTM C-613, and add the recovered filler weight to the dried thimble plus sample weight (W4).
- f. Calculate the dry resin content (resin solids) as follows:

$$\text{DRC, wt. \%} = 100 \left(1 - \frac{W4 - W1}{W}\right)$$

Where W = Devolatilized sample weight, gm.

W1 = Weight of dried thimble, gm.

W4 = Weight of thimble plus extracted sample, gm.

- g. Filler content can be calculated by utilizing the filler content in the resin mix before impregnation as follows:

$$\text{Filler content, wt. \%} = \frac{(\text{DRC}) (\text{FC})}{(100 - \text{FC})}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content of the resin mix

- h. Cloth content can be calculated as follows:

$$\text{Cloth content, wt. \%} = 100 - \text{DRC} - \text{FC}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content 4.6.2.2g

4.6.2.2.1 As an alternative method for determining filler content and cloth content, the following procedure may be used:

- a. Follow the procedures of 4.6.2.2 thru 4.6.2.2c.
- b. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, and allow the solvent to drain.
- c. Carefully open the thimble, remove the cloth and place the cloth in the bottom of a clean 250 ml beaker. Add 20-25 ml of ethyl alcohol and gently stir to remove any remaining filler.
- d. Transfer the liquid solution back to the original thimble, removing any visible fibers and keeping the fibers with the fabric.
- e. Rinse the fabric with ethyl alcohol several times to ensure complete removal of the filler from the fabric and add the rinses back to the original thimble. Make the final rinse acetone to remove all traces of ethyl alcohol.
- f. Dry the fabric in an oven for a minimum of 30 minutes at 325°F, cool in a desiccator to room temperature and reweigh to the nearest .001 gm (W5). This is the dry cloth weight.
- g. Close the top of the thimble and dry to a constant weight at 325 ± 5°F for 2 hours minimum in a forced air oven. Remove from the oven and immediately weigh to .001 gm (W6).
- h. Calculate the resin solids, filler content and cloth content as follows:

$$\text{Filler content, wt. \%} = \left(\frac{W6 - W1}{W} \right) \times 100$$

$$\text{Cloth content, wt. \%} = \left(\frac{W5}{W} \right) \times 100$$

$$\text{Resin solids content, wt. \%} = 100 - \left(\frac{W6 + W5 - W1}{W} \right)$$

Where W = devolatilized sample weight from 4.6.2.2

W1 = weight of dried thimble, gm.

W5 = weight of dried cloth, gm.

W6 = weight of thimble plus filler, gm.

4.6.2.3 Resin Flow. Resin flow of each sample shall be determined in accordance with the following:

- a. Cut four 4-inch by 4-inch \pm 1/8-inch squares across the width of the fabric. All squares shall be bias cut to eliminate fiber loss in testing. Stack the squares uniformly on each other to make a specimen.
- b. Weigh the specimen of four plies to the nearest 0.01 g and record as W1.
- c. Place the specimen between release film. Preheat the press to $325 \pm 10^\circ\text{F}$, position the specimen in the middle of the press plate and apply the press load of 150 ± 10 pounds per square inch gauge (psig) immediately. Press-load the specimen for a minimum of 10 minutes at $325 \pm 10^\circ\text{F}$.
- d. Remove the specimen from the press and cool to ambient temperature.
- e. Using the edge of a metal plate or knife, scrape off the resin flash to the original size of the specimen. Do not remove any reinforcement from the original dimensions.

NOTE: Include any fibers which may be displaced and scraped off during the process in the weighed back specimen weight (W2).

- f. Reweigh the specimen to the nearest 0.01 g and record as W2.
- g. Calculate the percent resin flow as follows:

$$\text{Percent flow} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g
W2 = final weight of specimen, g

4.6.2.4 Tack. Tack shall be determined in accordance with the following:

- a. Cut two 1.0 x 3" strips and place them lengthwise with a 1.0" overlap (warp to fill side) into a sheet of folded cellophane.
- b. Place the wrapped specimen onto a preheated 180°F hot plate and cover the overlapped area with a 2" x 2" x $\frac{1}{4}$ " metal block (weighing $125 \text{ gms.} \pm 5 \text{ gms.}$).
- c. Allow the specimen to heat for 30 ± 2 seconds.

- d. Remove the 125-gram block and immediately (within 5 seconds) place the specimen, with the cellophane intact, into a room temperature press and apply 50 ± 2 lbs. pressure to the 1" overlapped area for approximately $1\frac{1}{2}$ minutes, while the specimen cools. The press platens shall be covered with insulating non-metallic laminates to allow controlled cooling.
- e. Using an appropriate tensile testing device, apply a constant rate of loading to obtain sample failure within 5 to 10 seconds. Record and report the average load at failure of three determinations.

4.6.2.5 Sodium Content. Sodium content shall be determined in accordance with the following:

NOTE: Care should be exercised not to contaminate the sample with sodium. Samples should not be touched with bare hands.

- a. Take specimens which weigh approximately 3 g. Place test specimens in individual beakers and dry in an air circulating oven at $225 \pm 5^\circ\text{F}$ for a minimum of one hour.
- b. Remove the specimens from the oven and cool in a desiccator.
- c. Weigh approximately 2 g of each cooled specimen to the nearest 0.1 milligram (mg) into separate tared ceramic or platinum crucibles.
- d. Heat the crucibles in a muffle furnace at $600 \pm 15^\circ\text{C}$ for 16-18 hours or until constant weight is achieved.
- e. Remove the crucibles containing the ashed specimens, place in a desiccator and cool.
- f. Wash down the walls of each crucible with distilled water and add approximately 5 ml concentrated hydrochloric acid to each.
- g. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a 1 liter volumetric flask and dilute to volume with distilled water.
- h. Determine the sodium content of each flask by flame emission at 589 nanometers (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

4.6.3 Cured Material Tests.

4.6.3.1 Preparation of Test Panels. Cured material tests shall be determined on test panels prepared in accordance with the following, when referenced:

- a. Cut sufficient plies to make the specified panel thickness and minimum size required, maintaining and marking uniform orientation of cloth fiber direction.
- b. Wrap the stack of plies $1\frac{1}{2}$ times with a suitable release film such that the upper and lower surfaces and two sides are completely encased by the release film. The open ends allow for degassing and resin flow.
- c. Center the wrapped plies between two $\frac{1}{8}$ " thick smooth steel caul plates and center the caul plates in a press preheated to $325 \pm 10^\circ\text{F}$, starting the cure timer.
- d. Apply contact pressure (nominal 50-lb. load) and maintain for $30 \pm$ seconds. Increase the load rapidly to approximately 200-lb. load and immediately release the pressure until a clearance can be observed above the top caul plate.
- e. Immediately reapply contact pressure and "bump" the laminate as indicated above for one additional cycle.
- f. Increase the pressure slowly (over approximately 4 minutes) to 1000 ± 50 psi, controlling the rate of pressure application to minimize resin flow at the laminate edge.
- g. Maintain the pressure and temperature for 120 ± 15 minutes. Turn-off the platen heaters and allow the laminate to cool to below 150°F under pressure. Remove from the press, remove release film and resin flash. Do not use the outer $\frac{1}{2}$ " of the test panel for preparation of specimens, unless specifically directed.

4.6.3.2 Density. Density shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches by 5 inches minimum, prepared as per 4.6.3.1.
- b. Cut a specimen 1-inch by 1-inch by panel thickness at random from the test panel. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry 20 minutes, minimum, before testing.
- c. Weigh and record the weight of the specimen in air to the nearest 0.1 mg (W1).

d. Weigh the specimen in water at $73 \pm 3^\circ\text{F}$ and record the weight to the nearest 0.1 mg using a wire and sinker if required (W2).

e. Calculate density using water as follows:

$$\text{Density (g/cc)} = \frac{0.9975 W1}{(W1 + W3 - W2)}$$

Where: W1 = weight of specimen in air

W2 = weight of specimen, wire, and sinker suspended in water

W3 = weight of wire and sinker suspended in water to the same depth as in the W2 determination to the nearest 0.1 mg.

4.6.3.3 Residual Volatile Content. Residual volatile content shall be determined in accordance with the following:

a. Cut sufficient plies to produce a test panel 0.250 ± 0.050 inches thick and 4 inches by 4 inches, prepared as per 4.6.3.1.

b. Cut a specimen 1.0 inch by 1.0 inch by panel thickness from the test panel; do not machine the top and bottom molded surfaces. Wipe the specimen clean using MEK and allow to air dry 20 minutes minimum.

c. Place the specimen in a desiccator and desiccate for 18 hours minimum.

d. Weigh the specimen to the nearest 0.01 gram and record as W1.

e. Place the specimen in an air circulating oven, preheated and stabilized at $325 \pm 10^\circ\text{F}$. Maintain the specimen at a temperature of $325 \pm 10^\circ\text{F}$ for 4 hours.

f. Remove the specimen from the oven and cool in a desiccator for approximately 30 minutes or until the specimen reaches room temperature.

g. Reweigh the specimen to the nearest 0.01 gram. Record as W2.

h. Calculate the percent residual volatiles as follows:

$$\text{Residual volatile, \%} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = original weight of specimen, g

W2 = devolatilized weight of specimen, g

4.6.3.4 Resin Content. Resin content shall be determined in accordance with the following:

a. Prepare test specimens as follows:

- (1) Cut 32 rectangles (1/2 inch by 5 inches nominal) from a prepreg sample.
- (2) Stage (see 6.3.1) the 32 rectangles at $255 \pm 5^{\circ}\text{F}$ for approximately 20 minutes to reduce flow.
- (3) Using the 32 rectangles, mold a bar 1/2 inch by 1/2 inch by 5 inches.
- (4) Cure the specimen at $325 \pm 10^{\circ}\text{F}$ and 1000 ± 50 psi for 10 ± 1 minutes.

NOTE: If there is any resin flash, repeat and use a longer staging time.

- (5) Cut the bar into approximately three equal lengths to form three test specimens.

b. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry for 20 minutes, minimum, before testing.

c. Record specimen weight to the nearest 0.001 g (W).

d. Place the test specimen in the Vycor test tube and tightly attach the tube to the test apparatus (see Figure 2).

e. Start the vacuum pump and evacuate the system to less than two inches of Hg absolute. Check for air leaks by clamping off the hose to the vacuum pump and noting the manometer. The leak rate must be less than 0.20 inch of Hg per minute. Recheck joints until this level is achieved.

f. With the vacuum on and the pressure stabilized, place the Fisher burner under the specimen. Adjust flame area to completely cover the sample.

NOTE: A tube furnace maintained at $1500 \pm 25^{\circ}\text{F}$ may be used in place of the Fisher burner.

g. Continue pyrolysis for approximately 30 minutes or until the manometer or gauge reading is the same reading as it was prior to pyrolysis, whichever is longer.

If residue collects in the Vycor test tube, place the Fisher burners under those areas until the burnout is complete.

- h. When pyrolysis is complete, turn off burners and cool under vacuum until specimen is at room temperature.
- i. When the specimen is cool, turn off vacuum and carefully let air into the system. When pressure equilibrium is reached, remove specimen from test tube and weigh to the nearest 0.001 g (W1).
- j. Calculate dry resin content as follows:

$$\text{Dry resin content, percent} = \left(1.00 - \frac{W1}{W}\right) \times 100 \times K$$

Where: W1 = weight of specimen after pyrolysis, g

W2 = weight of specimen prior to pyrolysis, g

K = a constant as shown in Table V

TABLE V. K FACTORS

U.S. POLYMERIC FM 5834
TBD

4.6.3.5 Compressive Strength. Compressive strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches minimum by 5 inches minimum, prepared as per 4.6.3.1.
- b. Machine a specimen to the dimensions shown in Figure 3.
- c. Measure the width and thickness of the specimen to the nearest 0.001 inch in the gauge section area. Calculate and record the cross-sectional area.
- d. Place the specimen in the support jig (see Figure 4), centered so the ends project an even amount beyond both ends of the jig. Tighten finger tight only.

- e. Place the specimen and the support jig in the testing machine and align so that the specimen ends are parallel with the surface of the compression tool. Adjust the crosshead of the testing machine until it just contacts the top of the specimen.
- f. Set the speed control at an indicated crosshead rate of 0.050 inch per minute and start the machine.
- g. Record the maximum load carried by the specimen during the test.
- h. Calculate the compressive strength by dividing the maximum load by the original cross-section area.

4.6.3.6 Interlaminar Shear. Interlaminar shear strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 1.5 ± 0.25 inch thick across the plies by minimum dimensions of 4.0 by 4.0 inches.
- b. Cure at 1000 ± 50 psi and $325 \pm 10^\circ\text{F}$ for 120 ± 15 minutes.
- c. After cure, remove the panel from the press and allow the panel to cool to room temperature.
- d. Cut a specimen of sufficient size to produce a 0.375 inch diameter by 1.125 inch minimum length finished specimen with the length being perpendicular to the plies from the test panel.

NOTE: Do not use the outer 1/2 inch of the test panel for specimen preparation.

- e. Center the specimen to get the length of the cylindrical specimen perpendicular to the plies and machine the specimens to a diameter of $0.375 +0.000, -0.002$ inch diameter.
- f. Measure the diameter of the test specimen and calculate the cross-sectional area (A).
- g. Place the specimen in the 0.375 inch diameter three plate double-shear jig shown in Figure 5.
- h. Place the shear fixture in the testing machine, align, and pin the ends of the fixture.
- i. Set the machine at an indicated crosshead speed of 0.015 to 0.025 inch per minute and test.

j. Record the maximum load carried by the specimen during the test and record as L.

k. Calculations:

$$\text{Interlaminar shear (psi)} = \frac{L}{2A}$$

Where: L = the maximum load carried by the specimen during testing in pounds

A = the original cross-sectional area of the specimen in square inches.

4.6.3.7 Thermal Conductivity. Thermal conductivity shall be determined in accordance with ASTM C 177 or equivalent and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Sample thickness is dependent on the dimensions of the heating unit and shall be restricted to limit the theoretical error to 1%.
- c. Determine both cross ply and with ply thermal conductivity at 250°F. Faces of the specimen must be parallel to 1% of specimen thickness.

4.6.3.8 Coefficient of Thermal Expansion. The coefficient of thermal expansion (see 6.3.2) shall be determined in accordance with ASTM E 831 and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Determine the coefficient of thermal expansion in both the with ply or parallel, and cross ply or perpendicular direction of the specimens.

4.6.3.9 Flexural Strength. Flexural strength shall be determined in accordance with ASTM D 790 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each of the desired yarn orientations, prepared as per 4.6.3.1.
- b. Test per method I, procedure A with a span-to-depth ratio of 16 to 1 with three specimens for each sample.

4.6.3.10 Tensile Strength. Tensile strength shall be determined in accordance with ASTM D 638 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each desired yarn orientations, as per 4.6.3.1.
- b. Test with Type I specimens, with dimensions and tolerances for material thickness 0.28 or under, and testing speed of 0.2 in/min.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. The rolls shall be in sealed bags conforming to MIL-B-131, Type 1, Class 1 barrier material. Each broadgoods roll shall be separately wrapped with a 1-unit, minimum, dessicant bag conforming to MIL-D-3464 enclosed within each bag.

5.1.1 Broadgoods Shipping Container. The broadgoods shall be shipped in a container that can be opened from the top and will suspend both ends of the roll in the container.

5.2 Special Shipping Requirements. During shipment, the material temperature shall be maintained at or below 90°F. The ambient temperature to which the material is exposed during shipment shall be recorded with a temperature recording device provided by the procuring activity and placed with each lot shipment by the supplier.

5.3 Marking. Unless otherwise specified in the purchase document, each container and roll shall be marked for identification and shipment and shall include the following:

- a. Supplier lot number and material name
- b. Purchase document number
- c. Acceptance stock and lot number
- d. Number of rolls
- e. Roll net weight
- f. Container identification number
- g. Date of manufacture
- h. Mark each roll on inside of spool with supplier name, roll number, lot number and material name with ink stamp, stencil or adhesive label.

- i. This specification number and revision letter
- j. Mark each container on two opposite sides with the following:

**TEMPERATURE SENSITIVE MATERIAL
DO NOT EXPOSE TO TEMPERATURES EXCEEDING 90°F**

6. NOTES

- 6.1 Intended Use. The carbon cloth phenolic covered by this specification is intended for use in the manufacture of rocket motor nozzle components.
- 6.2 Ordering Data. Purchase documents should specify the following:
 - a. Title, number, and date of this specification
 - b. Special marking, packing, packaging, and shipping
 - c. Data retention requirements
 - d. Broadgoods roll width
 - e. Qualification sampling requirements (if required)
 - f. Number of copies of inspection and test data
 - g. Type of material: broadgoods or trimmed broadgoods
 - h. Acceptance stock and lot number
- 6.3 Definitions.
 - 6.3.1 Stage. A method of stabilizing and retaining the phenolic polymer in a solid state on the carbon cloth by temperature cycling.
 - 6.3.2 Coefficient of Thermal Expansion. The slope of the straight line intersecting the linear expansion curve at 75°F and 400°F expressed as change in length per unit length per °F.
- 6.4 Qualification. The procuring activity reserves the right to purchase only those products which have been previously tested and found to meet the requirements of this specification and which have proved satisfactory in one full scale nozzle static test. Prospective suppliers whose products have not been tested and found satisfactory are required to have their products tested in order that they may be considered eligible to be awarded contracts for the product covered by this specification.
- 6.5 Storage. After receipt of the material, the procuring activity is responsible for storage and for retesting to determine storage life extension.

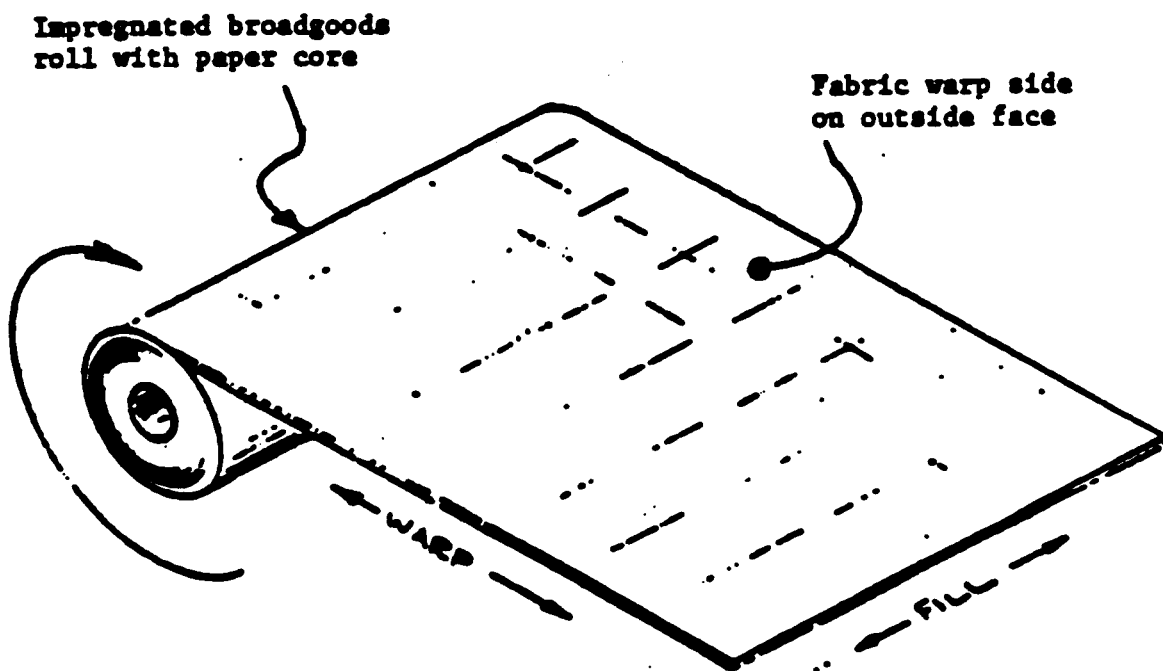


Figure 1. Requirements for Broadgoods Rolls

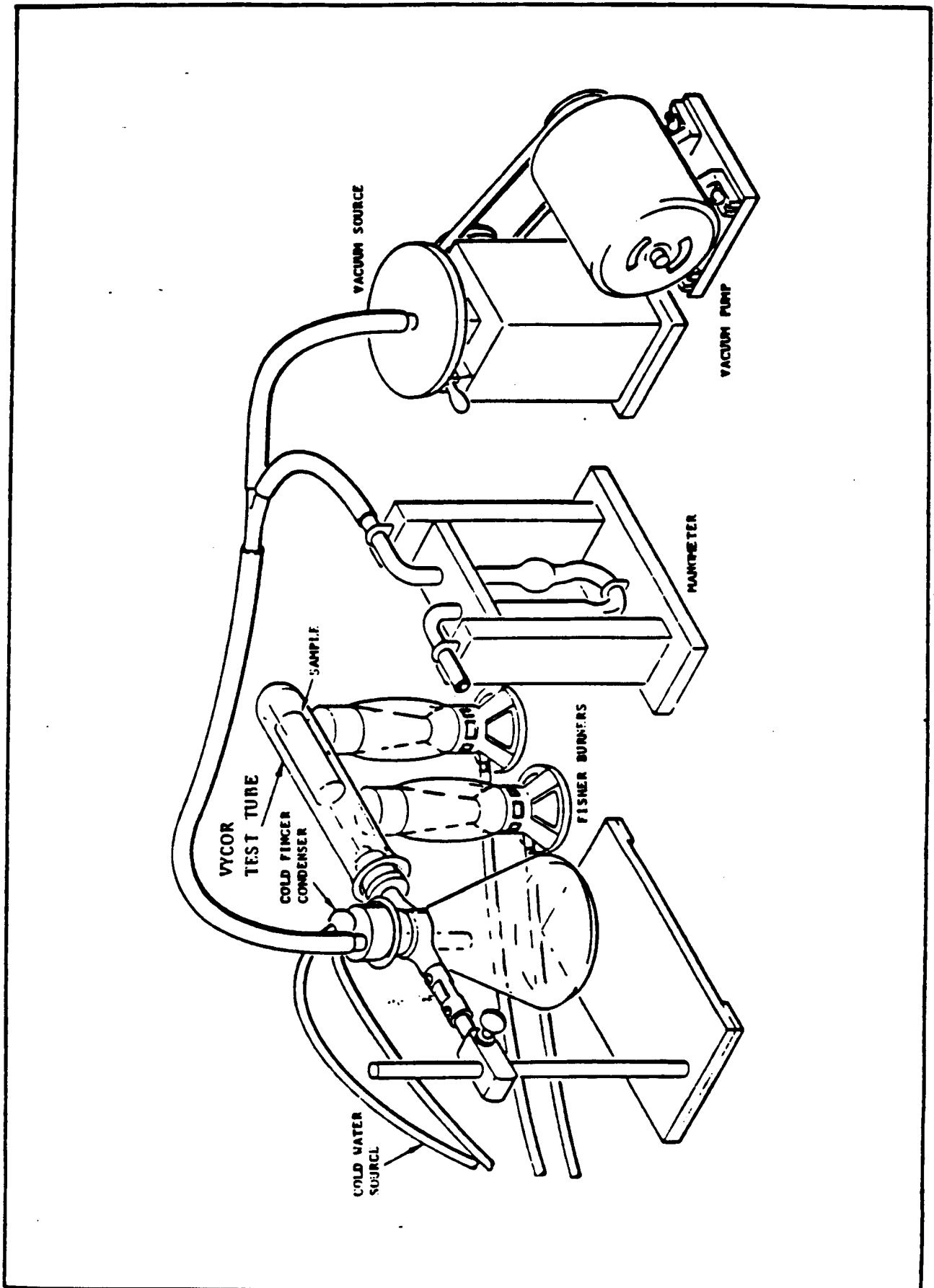
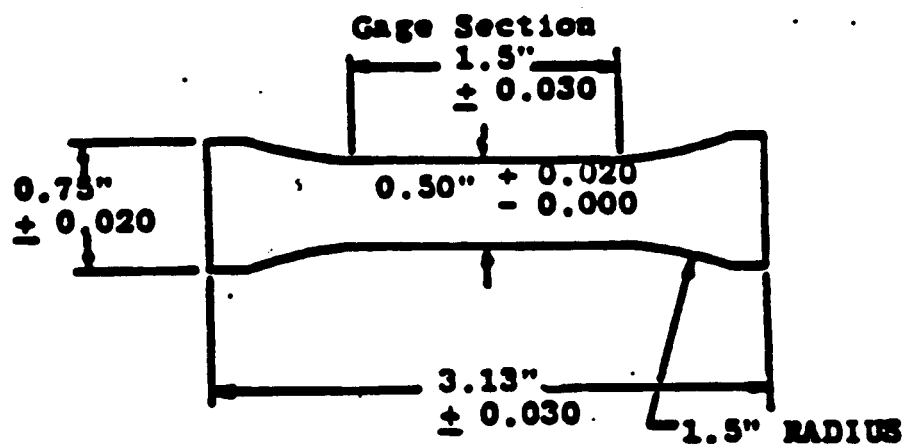
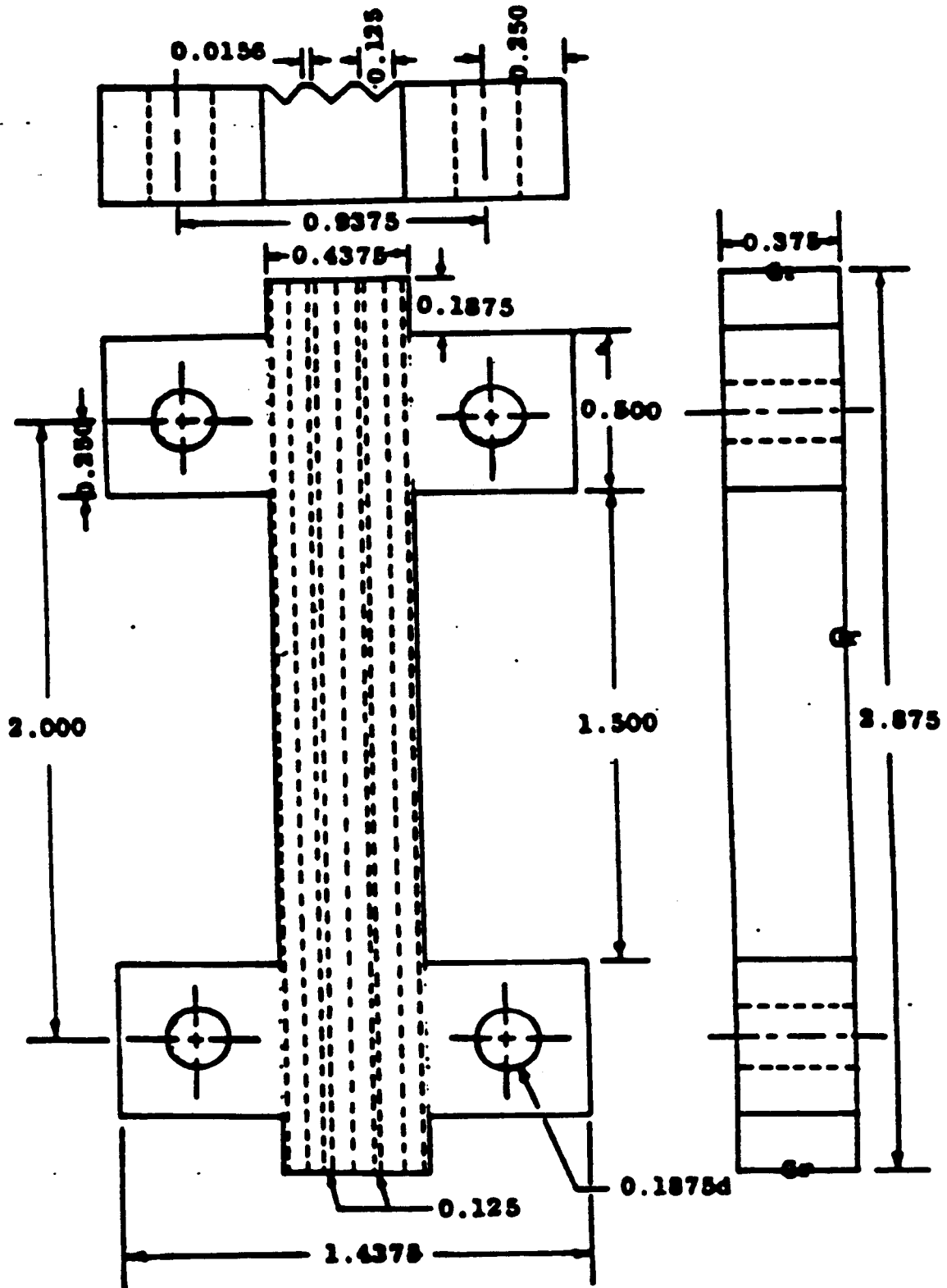


Figure 2. Resin Content Apparatus Setup



Test specimen thickness = 0.125 \pm 0.025 inch

Figure 3. Compressive Strength Test Specimen



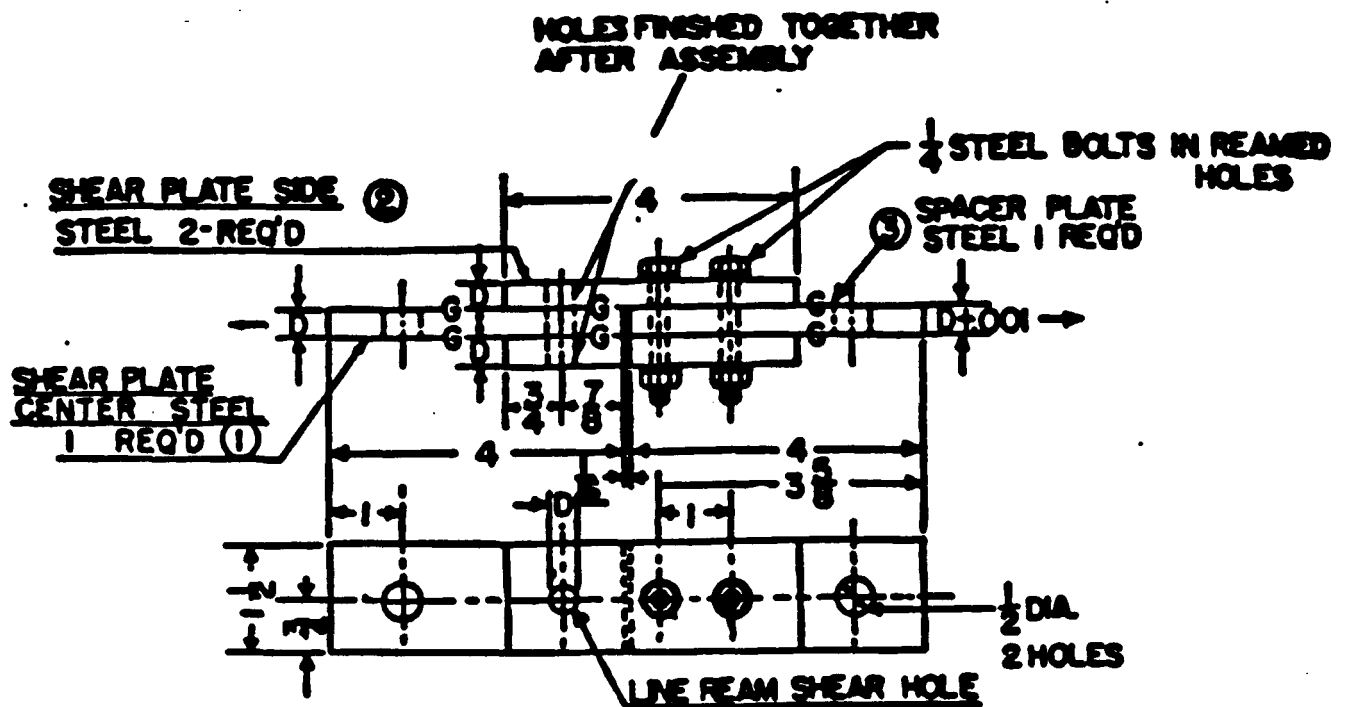
NOTE 1. - Cold rolled steel

NOTE 2. - Furnished four steel machine screws and nuts, round head, slotted, length 1-1/4 in.

NOTE 3. - Grind surfaces denoted "Gr"

Figure 4. Support Jig Details

ORIGINAL PAGE IS
OF POOR QUALITY



1 February 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

GRAPHITE CLOTH PHENOLIC, PREIMPREGNATED

1. SCOPE

1.1 Scope. This specification covers one type of graphite cloth reinforcement preimpregnated with a carbon filled phenolic resin.

1.1 Classification. The preimpregnated graphite cloth shall be of the following types, as specified (see 6.2h):

Type I - Broadgoods (Material tested for flow at 150 psi).

Type II - Broadgoods (Material tested for flow at 1000 psi).

2. APPLICABLE DOCUMENTS

2.1 Government Documents. The following documents of the issue in effect on date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

SPECIFICATIONS

Military

MIL-B-131 Barrier Materials, Watervapor-proof, Grease-proof, Flexible, Heat-sealable

MIL-D-3464 Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification

MIL-R-9299 Resin, Phenolic, Laminating

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Non-Government Documents. Unless otherwise specified, the following documents of the latest approved issues form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (USP-28) Filler, Carbon Black

MSFC-SPEC (WCA) Reinforcement, Graphite Cloth

MSFC-SPEC (USP-39A) Resin, Phenyladehyde

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812.)

American Society for Testing and Materials (ASTM)

ASTM C 177	Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate, Standard Test Method for
ASTM C 613	Resin Content of Carbon and Graphite Prpregs by Solvent Extraction, Standard Test Method for
ASTM D 638	Tensile Properties of Plastics, Standard Test Method for
ASTM D 790	Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials, Standard Test Methods for
ASTM E 831	Linear Thermal Expansion of Solid Materials by Thermodilatometry, Standard Test Method for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

- 3.1 Qualification. The material furnished under this specification shall be a product which has been tested and has passed the qualification inspection specified herein (see 4.3 and 6.4).
- 3.2 Material. The material shall be a graphite fabric reinforcement impregnated with a carbon filled phenolic resin and supplied as broadgoods. Broadgoods roll widths shall be 36 to 48 inches and shall be rolled onto the paper core with the warp side of the impregnated fabric on the outside face of the rolls (see Figure 1).

3.2.1 Resin. The resin shall conform to the requirements of MIL-R-9299, Grade A, and also to MSFC-SPEC (USP-39A).

3.2.2 Reinforcement. The reinforcement shall be a graphite fabric conforming to MSFC-SPEC (WCA).

3.2.3 Resin Filler. The resin filler shall be carbon powder conforming to MSFC-SPEC (USP-28).

3.3 Properties

3.3.1 Uncured Material. The properties of the uncured material shall be in accordance with Table I.

TABLE I. UNCURED MATERIAL PROPERTIES

PROPERTY	SPECIFICATION LIMITS		RETEST PROPERTIES <u>1/</u>	
	MINIMUM	MAXIMUM	MINIMUM	MAXIMUM
Cloth Content, %	48.0	58.0	----	----
Dry Resin Solids, %	28.0	37.0	----	----
Volatile Content, % Type I	---	5.5	----	6.0
Type II	---	3.5	----	4.0
Carbon Filler Content, %	12.0	18.0	----	----
Resin Flow, % Type I, <u>2/</u>	5.0	15.0	3.5	17.0
Type II	10.0	20.0	8.0	23.0
Tack, lbs. Type I	25	----	25	----
Type II	25	----	25	----
Sodium Content, ppm	----	100	----	----

1/ Procuring activity testing for extension of material shelf life after six months from date of supplier manufacture.

2/ Type I = 150 psi flow
Type II = 1000 psi flow

3.3.2 Cured Material. The properties of the cured material shall be in accordance with Table II.

TABLE II. CURED MATERIAL PHYSICAL AND MECHANICAL PROPERTIES
(AT ROOM TEMPERATURE UNLESS OTHERWISE SPECIFIED)

PROPERTY	LIMITS	
	MINIMUM	MAXIMUM
Density, grams per cubic centimeter (g/cc)	1.400	1.460
Residual Volatiles, percent	0.0	2.5
Resin Content, percent	28.0	35.0
Compressive Strength, psi (edgewise)		
warp direction	10,000	30,000
fill direction	8,000	24,000
Interlaminar Double Shear Strength, psi	2,200	4,000
Thermal Conductivity, Btu/ft-hr-degrees F		
at 250°F with ply	0.10	3.0
across ply	0.10	1.5
Coefficient of Thermal Expansion		
in/in-degrees F x 10 exp -6		
at 400°F across ply	2.0	15.0
with ply	0.0	5.0
Flexural Strength, psi		
warp direction	20,000	35,000
fill direction	16,000	26,000
Tensile Strength, psi		
warp direction	14,000	30,000
fill direction	5,000	20,000

3.4 Age of Material. The material shall be shipped within 3 months of manufacturing date and shall be tested and certified within 60 days of shipment.

3.5 Process of Manufacture. Any change in process or material after initial qualification testing as required by this specification shall be cause to require requalification.

- 3.6 Storage Life. The material shall have a storage life of six months after date of manufacture when stored at 50°F maximum in sealed plastic bags with a 1-unit minimum desiccant bag conforming to MIL-D-3464. After date of receipt of the broadgoods, the maximum cumulative out-of-storage time shall be 30 days at 85°F maximum. Storage life requirements shall apply until the material has been completely cured.
- 3.6.1 Storage Life Extension. For the convenience of the procuring activity, the storage life of broadgoods may be extended for three additional 6-month periods provided the material, upon retest (see 6.5), conforms to the retest requirement for resin flow, volatile content, and tack in Table I. The maximum allowable cumulative storage life of broadgoods shall not exceed 24 months from the date of manufacture of the original broadgoods lot. Each individual roll of broadgoods must be tested for storage life extension.
- 3.6.2 Conditioning for Storage Life Extension Retest. Material which fails to meet the allowable volatile, tack and resin flow retest requirements may be conditioned to extend the storage life by subjecting the rolls which failed to meet the requirements to the following:
- Place broadgoods material in a vacuum chamber supported through core(s) to prevent material damage.
 - Expose material to a vacuum of less than one inch of mercury (Hg) absolute and a temperature of 60-90°F for a period of 7-9 days.
 - After completion of the drying process, obtain samples from outside and inside of each roll and retest for volatile content resin flow and tack.
- Failure of any of the conditioned rolls to meet the retest requirements of Table I shall require the roll to be scrapped.
- 3.6.3 Out-of-Storage Time Extension - The 30-day out-of-storage time at 85°F maximum may be extended for one additional 30-day period provided that each roll and the average of three tests has met the retest requirements for volatile content, resin flow and tack specified in Table I.
- 3.6.4 Preparation and Storage of Samples Selected for Retest. Each lot of samples shall be promptly put into an individual plastic bag, sealed, properly labeled, and stored with the broadgoods it represents. Prior to testing and removal from the plastic bag, the samples shall be conditioned at 60-80°F for two hours minimum. The samples shall be tested within 72 hours after removal from plastic bags and within 14 days after removal from the broadgoods rolls.

3.7 Workmanship

- 3.7.1 Broadgoods. The material shall have no holes, tears, non-uniform weave, resin build-up, contaminants, or other defects which would render more than 5 percent of any roll unsuitable for the intended use.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test results conform to prescribed requirements.
- 4.2 Classification of Inspections. The inspection requirements specified herein are classified as follows:
- a. Qualification inspection (see 4.3)
 - b. Quality conformance inspection (see 4.4).
- 4.3 Qualification Inspection. Three rolls of broadgoods from the first production lot (see 4.4.1.1) shall be subjected to all examinations and tests specified in Table III. The lots subjected to qualification inspection shall be representative of the manufacturing production from the proposed production facility.

TABLE III. QUALIFICATION INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5

TABLE III. QUALIFICATION INSPECTION (CONT'D.)

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Residual Volatiles	3.3.2	4.6.3.3
Resin Content	3.3.2	4.6.3.4
Compressive Strength	3.3.2	4.6.3.5
Interlaminar Shear	3.3.2	4.6.3.6
Thermal Conductivity	3.3.2	4.6.3.7
Coefficient of Thermal Expansion	3.3.2	4.6.3.8
Flexural Strength	3.3.2	4.6.3.9
Tensile Strength	3.3.2	4.6.3.10

4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of all the examinations and tests specified in Table IV performed on each lot.

TABLE IV. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TEST	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Resin Content	3.3.2	4.6.3.4
Compressive Strength (warp direction only)	3.3.2	4.6.3.5

4.4.1 Sampling. Each lot of graphite cloth phenolic shall be sampled for inspection as follows:

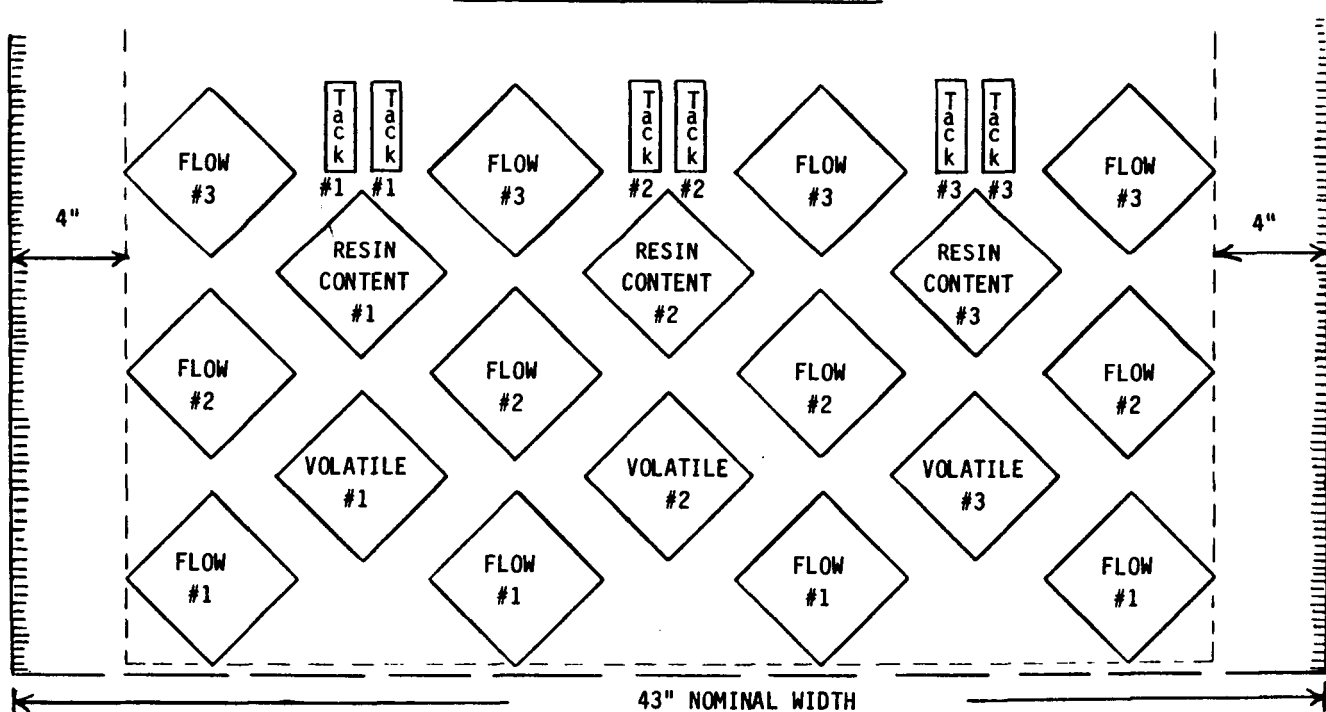
4.4.1.1 Lot. A manufacturing lot of preimpregnated material shall consist of material which is impregnated in one continuous production run, using one batch of the impregnator's resin and additives mixed in one vessel at one time.

4.4.1.1.1 Lot Size. A lot of graphite cloth phenolic shall consist of not more than 15,000 pounds.

4.4.1.2 Lot Acceptance Test Samples.

4.4.1.2.1 Sampling for Uncured Material Properties. Each sample shall be of sufficient size to perform all of the uncured material inspections and tests. Each sample shall be marked with the roll number, lot number, and the location in the roll from which the sample was taken. Each broadgoods roll in the lot shall be sampled at the exposed end of the roll, as shipped.

TYPICAL UNCURED MATERIAL SAMPLING



- 4.4.1.2.2 Sampling for Cured Material Properties. A sample of sufficient size to perform all the cured material inspections and tests specified herein shall be taken from the exposed end of each broadgoods roll selected at random from each lot. The number of rolls to be selected shall be determined as follows:

NUMBER OF ROLLS IN LOT	NUMBER OR ROLLS TO BE SELECTED FOR SAMPLING
1 to 2	All
3 to 40	3
41 to 65	4
66 to 110	5

Failure of a roll sample to conform to the requirements of this specification shall be cause to withdraw that roll and shall require that all other rolls in that lot be tested for only those material properties which failed to meet specification requirements.

- 4.5 Test Requirements. Three tests shall be performed for each of the material properties specified herein on the samples taken from each broadgoods roll sampled. The average values of the test results and at least two of the three individual test values shall be used to determine conformance to the requirements of this specification.

- 4.5.1 Test Conditions. Unless otherwise specified, all tests shall be performed under ambient conditions.

- 4.5.1.1 Supplier Retest. When the average test value of any material property fails to conform to Tables I and II and the cause of the failure can be attributed to improper preparation of the specimen or testing machine anomaly, new tests shall be conducted on a sample from the same broadgoods roll from which the failed specimens were removed.

- 4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20 percent shall be used.

4.6.1 Visual Examination. Each roll of preimpregnated cloth shall be visually examined for conformance to 3.7 and Section 5 before supplier shipment.

4.6.1.1 Visual Examination for Conformance to Special Shipping and Marking Requirements. Temperature record charts of the ambient temperature to which the material was exposed during shipment shall be examined for conformance to 5.2. Marking shall be visually inspected for conformance to 5.3.

4.6.2 Uncured Material Tests.

4.6.2.1 Volatile Content. The volatile content of each sample of the uncured material shall be determined in accordance with the following:

- a. Cut a 16 ± 2 square inch specimen.
- b. Weigh the specimen to the nearest 0.01 gram (g) (W1).
- c. Place specimen in a recirculating oven preheated and stabilized to $325 \pm 10^\circ\text{F}$ for 10 ± 1 minutes. Specimens should be placed to get adequate air circulation.
- d. Remove the specimen and within one (1) minute, weigh to the nearest 0.01 g (W2).
- e. Calculate percent volatiles as follows:

$$\text{Percent volatiles} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g

W2 = final weight of specimen, g

4.6.2.2 Resin Solids, Filler Content, and Cloth Content. Resin solids, shall be determined for each sample in accordance with the soxhlet extraction method specified in ASTM C 613 and the following:

- a. Precondition a paper thimble at $325 \pm 5^\circ\text{F}$ for 120 minutes. Weigh immediately to .001 gm (W1) and store in a desiccator with the recorded weight until ready to use.
- b. Reweigh the thimble immediately before addition of the sample (W2) so an accurate sample weight is established. Place a bias cut 2.0 x 4.0 inch prepreg sample into the thimble and weigh to .001 gm (W3).

- c. Using the volatile content obtained in 4.6.2.1e, calculate the devolatilized sample weight by using the formula:

$$W = \text{devolatilized sample weight} = (W3 - W2) \times \left(1 - \frac{V}{100}\right)$$

Where W2 = Thimble weight immediately before sample addition, gm.

W3 = Thimble weight plus sample, gm.

V = Volatile content, wt. %

- d. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, allow the solvent to drain, and dry to constant weight at $325 \pm 5^\circ\text{F}$ for 2 hours minimum in a forced air oven. Remove from the oven in sets of three, and immediately weigh to .001 gm (W4).
- e. If filler has appeared in the extract, determine the filler weight lost by the filtering and crucible technique in ASTM C-613, and add the recovered filler weight to the dried thimble plus sample weight (W4).
- f. Calculate the dry resin content (resin solids) as follows:

$$\text{DRC, wt. \%} = 100 \left(1 - \frac{W4 - W1}{W}\right)$$

Where W = Devolatilized sample weight, gm.

W1 = Weight of dried thimble, gm.

W4 = Weight of thimble plus extracted sample, gm.

- g. Filler content can be calculated by utilizing the filler content in the resin mix before impregnation as follows:

$$\text{Filler content, wt. \%} = \frac{(\text{DRC})(\text{FC})}{(100 - \text{FC})}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content of the resin mix

- h. Cloth content can be calculated as follows:

$$\text{Cloth content, wt. \%} = 100 - \text{DRC} - \text{FC}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content 4.6.2.2g

4.6.2.2.1 As an alternative method for determining filler content and cloth content, the following procedure may be used:

- a. Follow the procedures of 4.6.2.2 thru 4.6.2.2c.
- b. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, and allow the solvent to drain.
- c. Carefully open the thimble, remove the cloth and place the cloth in the bottom of a clean 250 ml beaker. Add 20-25 ml of ethyl alcohol and gently stir to remove any remaining filler.
- d. Transfer the liquid solution back to the original thimble, removing any visible fibers and keeping the fibers with the fabric.
- e. Rinse the fabric with ethyl alcohol several times to ensure complete removal of the filler from the fabric and add the rinses back to the original thimble. Make the final rinse acetone to remove all traces of ethyl alcohol.
- f. Dry the fabric in an oven for a minimum of 30 minutes at 325°F, cool in a desiccator to room temperature and reweigh to the nearest .001 gm (W5). This is the dry cloth weight.
- g. Close the top of the thimble and dry to a constant weight at 325 ± 5°F for 2 hours minimum in a forced air oven. Remove from the oven and immediately weigh to .001 gm (W6).
- h. Calculate the resin solids, filler content and cloth content as follows:

$$\text{Filler content, wt. \%} = \left(\frac{W6 - W1}{W} \right) \times 100$$

$$\text{Cloth content, wt. \%} = \left(\frac{W5}{W} \right) \times 100$$

$$\text{Resin solids content, wt. \%} = 100 - \left(\frac{W6 + W5 - W1}{W} \right)$$

Where W = devolatilized sample weight from 4.6.2.2

W1 = weight of dried thimble, gm.

W5 = weight of dried cloth, gm.

W6 = weight of thimble plus filler, gm.

4.6.2.3 Resin Flow. Resin flow of each sample shall be determined in accordance with the following:

- a. Cut four 4-inch by 4-inch \pm 1/8-inch squares across the width of the fabric. All squares shall be bias cut to eliminate fiber loss in testing. Stack the squares uniformly on each other to make a specimen.
- b. Weigh the specimen of four plies to the nearest 0.01 g and record as W1.
- c. Place the specimen between release film. Preheat the press to $325 \pm 10^\circ\text{F}$, and position the specimen in the middle of the press plate. For Type I material, apply a press load of 150 ± 10 pounds per square inch gauge (psig) immediately. For Type II material, apply a press load of 1000 ± 50 psig. Press load the specimen for a minimum of 10 minutes @ $375 \pm 10^\circ\text{F}$.
- d. Remove the specimen from the press and cool to ambient temperature.
- e. Using the edge of a metal plate or knife, scrape off the resin flash to the original size of the specimen. Do not remove any reinforcement from the original dimensions.

NOTE: Include any fibers which may be displaced and scraped off during the process in the weighed back specimen weight (W2).

- f. Reweigh the specimen to the nearest 0.01 g and record as W2.
- g. Calculate the percent resin flow as follows:

$$\text{Percent flow} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g
W2 = final weight of specimen, g

4.6.2.4 Tack. Tack shall be determined in accordance with the following:

- a. Cut two 1.0 x 4" strips and place them lengthwise with a 1.0" overlap (wrap to fill side) into a sheet of folded cellophane.
- b. Place the wrapped specimen onto a preheated $190 \pm 5^\circ\text{F}$ press. Close the press to 50 psi as rapidly as possible, and hold this pressure for 60 seconds.
- c. Remove the specimen from the press, and place between two 1/8 inch thick room temperature plates and allow to cool to room temperature.

- d. Using an appropriate tensile testing device, apply a constant rate of loading to obtain sample failure within 5 to 10 seconds. Record and report the average load at failure of three determinations.

4.6.2.5 Sodium Content. Sodium content shall be determined in accordance with the following:

NOTE: Care should be exercised not to contaminate the sample with sodium. Samples should not be touched with bare hands.

- a. Take specimens which weigh approximately 3 g. Place test specimens in individual beakers and dry in an air circulating oven at $225 \pm 5^{\circ}\text{F}$ for a minimum of one hour.
- b. Remove the specimens from the oven and cool in a desiccator.
- c. Weigh approximately 2 g of each cooled specimen to the nearest 0.1 milligram (mg) into separate tared ceramic or platinum crucibles.
- d. Heat the crucibles in a muffle furnace at $600 \pm 15^{\circ}\text{C}$ for 16-18 hours or until constant weight is achieved.
- e. Remove the crucibles containing the ashed specimens, place in a desiccator and cool.
- f. Wash down the walls of each crucible with distilled water and add approximately 5 ml concentrated hydrochloric acid to each.
- g. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a 1 liter volumetric flask and dilute to volume with distilled water.
- h. Determine the sodium content of each flask by flame emission at 589 nanometers (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

4.6.3 Cured Material Tests.

4.6.3.1 Preparation of Test Panels. Cured material tests shall be determined on test panels prepared in accordance with the following, when referenced:

- a. Cut sufficient plies to make the specified panel thickness and minimum size required, maintaining and marking uniform orientation of cloth fiber direction.
- b. Wrap the stack of plies $1\frac{1}{2}$ times with a suitable release film such that the upper and lower surfaces and two sides are completely encased by the release film. The open ends allow for degassing and resin flow.
- c. Center the wrapped plies between two $\frac{1}{8}$ " thick smooth steel caul plates and center the caul plates in a press preheated to $325 \pm 10^\circ\text{F}$, starting the cure timer.
- d. Apply contact pressure (nominal 50-lb. load) and maintain for $30 \pm$ seconds. Increase the load rapidly to approximately 200-lb. load and immediately release the pressure until a clearance can be observed above the top caul plate.
- e. Immediately reapply contact pressure and "bump" the laminate as indicated above for one additional cycle.
- f. Increase the pressure slowly (over approximately 4 minutes) to 1000 ± 50 psi, controlling the rate of pressure application to minimize resin flow at the laminate edge.
- g. Maintain the pressure and temperature for 120 ± 15 minutes. Turn-off the platen heaters and allow the laminate to cool to below 150°F under pressure. Remove from the press, remove release film and resin flash. Do not use the outer $\frac{1}{2}$ " of the test panel for preparation of specimens, unless specifically directed.

4.6.3.2 Density. Density shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches by 5 inches minimum, prepared as per 4.6.3.1.
- b. Cut a specimen 1-inch by 1-inch by panel thickness at random from the test panel. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry 20 minutes, minimum, before testing.
- c. Weigh and record the weight of the specimen in air to the nearest 0.1 mg (W1).

- d. Weigh the specimen in water at $73 \pm 3^\circ\text{F}$ and record the weight to the nearest 0.1 mg using a wire and sinker if required (W2).

- e. Calculate density using water as follows:

$$\text{Density (g/cc)} = \frac{0.9975 W1}{(W1 + W3 - W2)}$$

Where: W1 = weight of specimen in air

W2 = weight of specimen, wire, and sinker suspended in water

W3 = weight of wire and sinker suspended in water to the same depth as in the W2 determination to the nearest 0.1 mg.

4.6.3.3 Residual Volatile Content. Residual volatile content shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.250 ± 0.050 inches thick and 4 inches by 4 inches, prepared as per 4.6.3.1.
- b. Cut a specimen 1.0 inch by 1.0 inch by panel thickness from the test panel; do not machine the top and bottom molded surfaces. Wipe the specimen clean using MEK and allow to air dry 20 minutes minimum.
- c. Place the specimen in a desiccator and desiccate for 18 hours minimum.
- d. Weigh the specimen to the nearest 0.01 gram and record as W1.
- e. Place the specimen in an air circulating oven, preheated and stabilized at $325 \pm 10^\circ\text{F}$. Maintain the specimen at a temperature of $325 \pm 10^\circ\text{F}$ for 4 hours.
- f. Remove the specimen from the oven and cool in a desiccator for approximately 30 minutes or until the specimen reaches room temperature.
- g. Reweigh the specimen to the nearest 0.01 gram. Record as W2.
- h. Calculate the percent residual volatiles as follows:

$$\text{Residual volatile, \%} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = original weight of specimen, g

W2 = devolatilized weight of specimen, g

4.6.3.4 Resin Content. Resin content shall be determined in accordance with the following:

a. Prepare test specimens as follows:

- (1) Cut 32 rectangles (1/2 inch by 5 inches nominal) from a prepreg sample.
- (2) Stage (see 6.3.1) the 32 rectangles at $255 \pm 5^{\circ}\text{F}$ for approximately 20 minutes to reduce flow.
- (3) Using the 32 rectangles, mold a bar 1/2 inch by 1/2 inch by 5 inches.
- (4) Cure the specimen at $325 \pm 10^{\circ}\text{F}$ and 1000 ± 50 psi for 10 ± 1 minutes.

NOTE: If there is any resin flash, repeat and use a longer staging time.

- (5) Cut the bar into approximately three equal lengths to form three test specimens.

b. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry for 20 minutes, minimum, before testing.

c. Record specimen weight to the nearest 0.001 g (W).

d. Place the test specimen in the Vycor test tube and tightly attach the tube to the test apparatus (see Figure 2).

e. Start the vacuum pump and evacuate the system to less than two inches of Hg absolute. Check for air leaks by clamping off the hose to the vacuum pump and noting the manometer. The leak rate must be less than 0.20 inch of Hg per minute. Recheck joints until this level is achieved.

f. With the vacuum on and the pressure stabilized, place the Fisher burner under the specimen. Adjust flame area to completely cover the sample.

NOTE: A tube furnace maintained at $1500 \pm 25^{\circ}\text{F}$ may be used in place of the Fisher burner.

g. Continue pyrolysis for approximately 30 minutes or until the manometer or gauge reading is the same reading as it was prior to pyrolysis, whichever is longer.

NOTE: If residue collects in the Vycor test tube, place the Fisher burners under those areas until the burnout is complete.

- h. When pyrolysis is complete, turn off burners and cool under vacuum until specimen is at room temperature.
- i. When the specimen is cool, turn off vacuum and carefully let air into the system. When pressure equilibrium is reached, remove specimen from test tube and weigh to the nearest 0.001 g (W1).
- j. Calculate dry resin content as follows:

$$\text{Dry resin content, percent} = \left(1.00 - \frac{W1}{W}\right) \times 100 \times K$$

Where: W1 = weight of specimen after pyrolysis, g

W2 = weight of specimen prior to pyrolysis, g

K = a constant as shown in Table V

TABLE V. K FACTORS

U.S. POLYMERIC FM 5064J
1.800

4.6.3.5 Compressive Strength. Compressive strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches minimum by 5 inches minimum, prepared as per 4.6.3.1.
- b. Machine a specimen to the dimensions shown in Figure 3.
- c. Measure the width and thickness of the specimen to the nearest 0.001 inch in the gauge section area. Calculate and record the cross-sectional area.
- d. Place the specimen in the support jig (see Figure 4), centered so the ends project an even amount beyond both ends of the jig. Tighten finger tight only.

- e. Place the specimen and the support jig in the testing machine and align so that the specimen ends are parallel with the surface of the compression tool. Adjust the crosshead of the testing machine until it just contacts the top of the specimen.
- f. Set the speed control at an indicated crosshead rate of 0.050 inch per minute and start the machine.
- g. Record the maximum load carried by the specimen during the test.
- h. Calculate the compressive strength by dividing the maximum load by the original cross-section area.

4.6.3.6 Interlaminar Shear. Interlaminar shear strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 1.5 ± 0.25 inch thick across the plies by minimum dimensions of 4.0 by 4.0 inches.
- b. Cure at 1000 ± 50 psi and $325 \pm 10^\circ\text{F}$ for 120 ± 15 minutes.
- c. After cure, remove the panel from the press and allow the panel to cool to room temperature.
- d. Cut a specimen of sufficient size to produce a 0.375 inch diameter by 1.125 inch minimum length finished specimen with the length being perpendicular to the plies from the test panel.

NOTE: Do not use the outer 1/2 inch of the test panel for specimen preparation.

- e. Center the specimen to get the length of the cylindrical specimen perpendicular to the plies and machine the specimens to a diameter of $0.375 +0.000, -0.002$ inch diameter.
- f. Measure the diameter of the test specimen and calculate the cross-sectional area (A).
- g. Place the specimen in the 0.375 inch diameter three plate double-shear jig shown in Figure 5.
- h. Place the shear fixture in the testing machine, align, and pin the ends of the fixture.
- i. Set the machine at an indicated crosshead speed of 0.015 to 0.025 inch per minute and test.

j. Record the maximum load carried by the specimen during the test and record as L.

k. Calculations:

$$\text{Interlaminar shear (psi)} = \frac{L}{2A}$$

Where: L = the maximum load carried by the specimen during testing in pounds

A = the original cross-sectional area of the specimen in square inches.

4.6.3.7 Thermal Conductivity. Thermal conductivity shall be determined in accordance with ASTM C 177 or equivalent and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Sample thickness is dependent on the dimensions of the heating unit and shall be restricted to limit the theoretical error to 1%.
- c. Determine both cross ply and with ply thermal conductivity at 250°F. Faces of the specimen must be parallel to 1% of specimen thickness.

4.6.3.8 Coefficient of Thermal Expansion. The coefficient of thermal expansion (see 6.3.2) shall be determined in accordance with ASTM E 831 and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Determine the coefficient of thermal expansion in both the with ply or parallel, and cross ply or perpendicular direction of the specimens.

4.6.3.9 Flexural Strength. Flexural strength shall be determined in accordance with ASTM D 790 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each of the desired yarn orientations, prepared as per 4.6.3.1.
- b. Test per method I, procedure A with a span-to-depth ratio of 16 to 1 with three specimens for each sample.

4.6.3.10 Tensile Strength. Tensile strength shall be determined in accordance with ASTM D 638 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each desired yarn orientations, as per 4.6.3.1.
- b. Test with Type I specimens, with dimensions and tolerances for material thickness 0.28 or under, and testing speed of 0.2 in/min.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. The rolls shall be in sealed bags conforming to MIL-B-131, Type 1, Class 1 barrier material. Each broadgoods roll shall be separately wrapped with a 1-unit, minimum, dessicant bag conforming to MIL-D-3464 enclosed within each bag.

5.1.1 Broadgoods Shipping Container. The broadgoods shall be shipped in a container that can be opened from the top and will suspend both ends of the roll in the container.

5.2 Special Shipping Requirements. During shipment, the material temperature shall be maintained at or below 90°F. The ambient temperature to which the material is exposed during shipment shall be recorded with a temperature recording device provided by the procuring activity and placed with each lot shipment by the supplier.

5.3 Marking. Unless otherwise specified in the purchase document, each container and roll shall be marked for identification and shipment and shall include the following:

- a. Supplier lot number and material name and type number
- b. Purchase document number
- c. Acceptance stock and lot number
- d. Number of rolls
- e. Roll net weight
- f. Container identification number
- g. Date of manufacture
- h. Mark each roll on inside of spool with supplier name, roll number, lot number and material name with ink stamp, stencil or adhesive label.

- i. This specification number and revision letter
- j. Mark each container on two opposite sides with the following:

**TEMPERATURE SENSITIVE MATERIAL
DO NOT EXPOSE TO TEMPERATURES EXCEEDING 90°F**

6. NOTES

- 6.1 Intended Use. The graphite cloth phenolic covered by this specification is intended for use in the manufacture of rocket motor nozzle components.
- 6.2 Ordering Data. Purchase documents should specify the following:
 - a. Title, number, and date of this specification
 - b. Special marking, packing, packaging, and shipping
 - c. Data retention requirements
 - d. Broadgoods roll width
 - e. Qualification sampling requirements (if required)
 - f. Number of copies of inspection and test data
 - g. Type of material: broadgoods or trimmed broadgoods
 - h. Material type (see 1.2)
 - i. Acceptance stock and lot number
- 6.3 Definitions.
 - 6.3.1 Stage. A method of stabilizing and retaining the phenolic polymer in a solid state on the graphite cloth by temperature cycling.
 - 6.3.2 Coefficient of Thermal Expansion. The slope of the straight line intersecting the linear expansion curve at 75°F and 400°F expressed as change in length per unit length per °F.
- 6.4 Qualification. The procuring activity reserves the right to purchase only those products which have been previously tested and found to meet the requirements of this specification and which have proved satisfactory in one full scale nozzle static test. Prospective suppliers whose products have not been tested and found satisfactory are required to have their products tested in order that they may be considered eligible to be awarded contracts for the product covered by this specification.
- 6.5 Storage. After receipt of the material, the procuring activity is responsible for storage and for retesting to determine storage life extension.

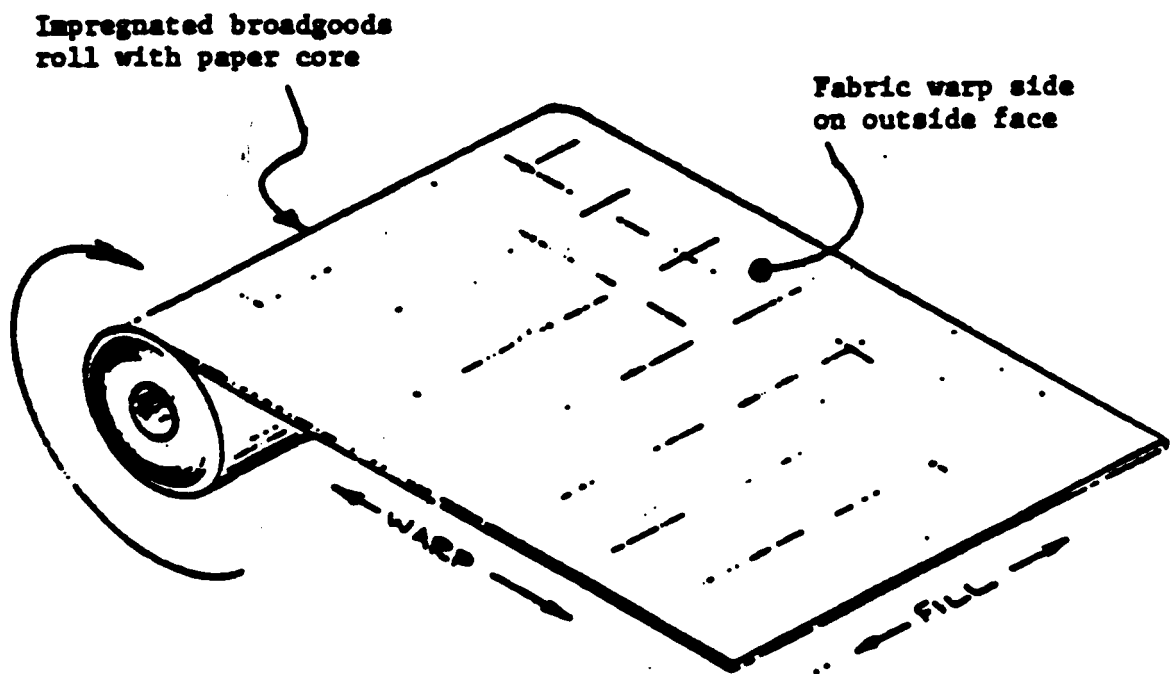


Figure 1. Requirements for Broadgoods Rolls

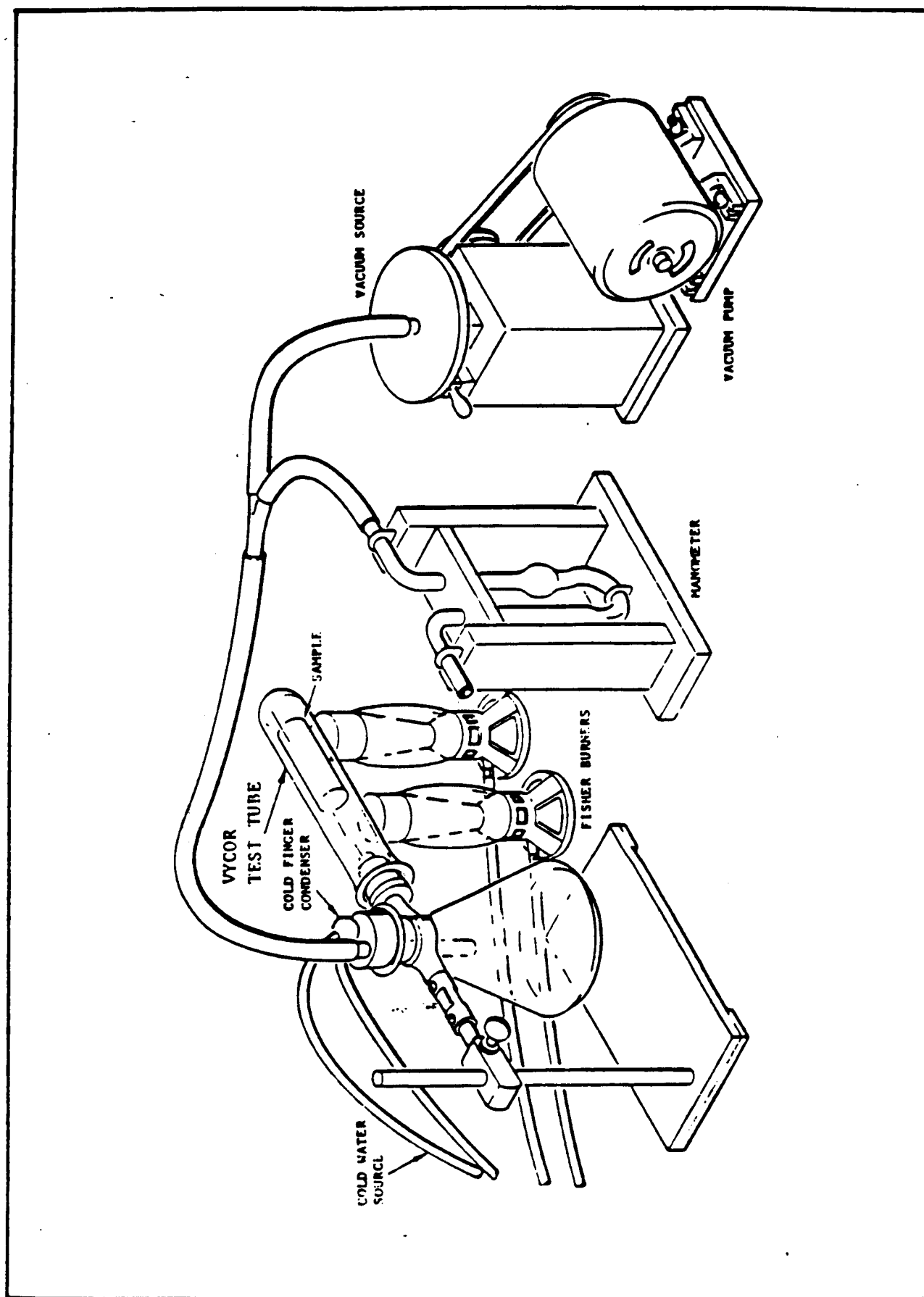
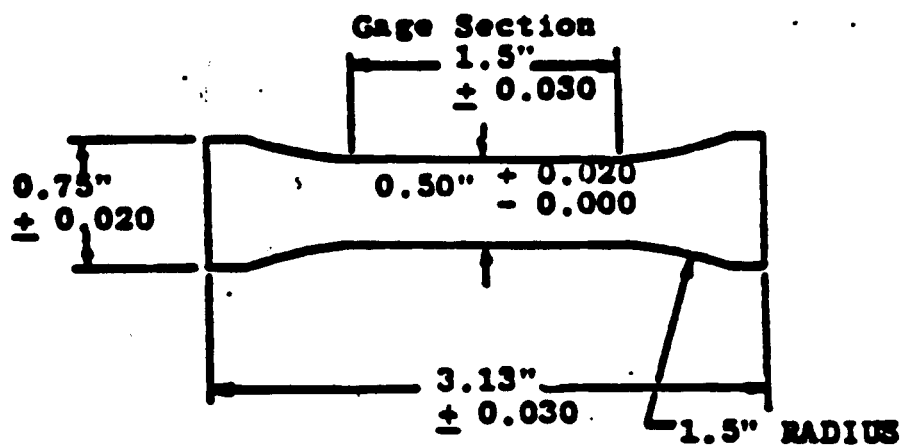
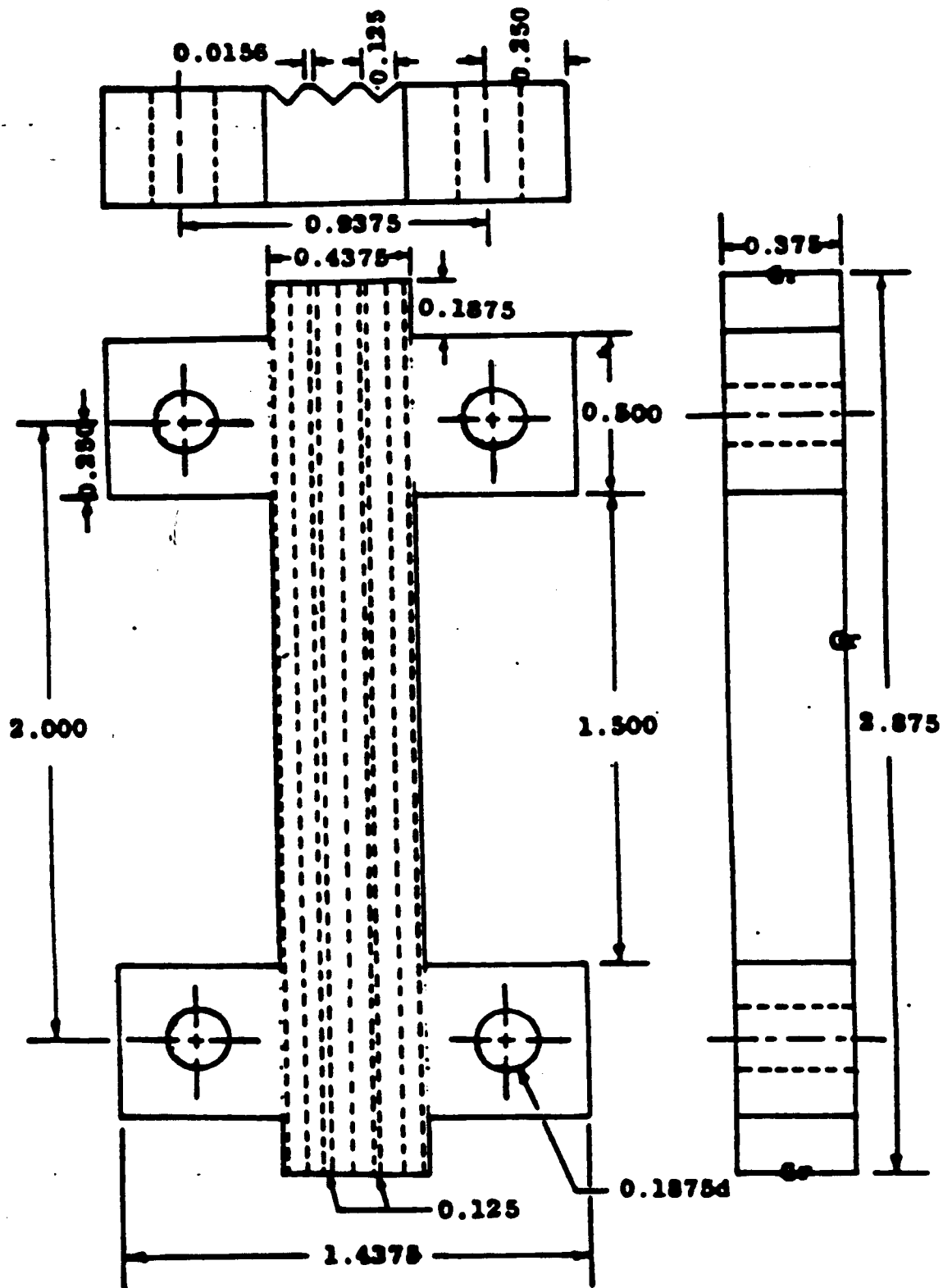


Figure 2. Resin Content Apparatus Setup



Test specimen thickness = 0.125 ± 0.025 inch

Figure 3. Compressive Strength Test Specimen



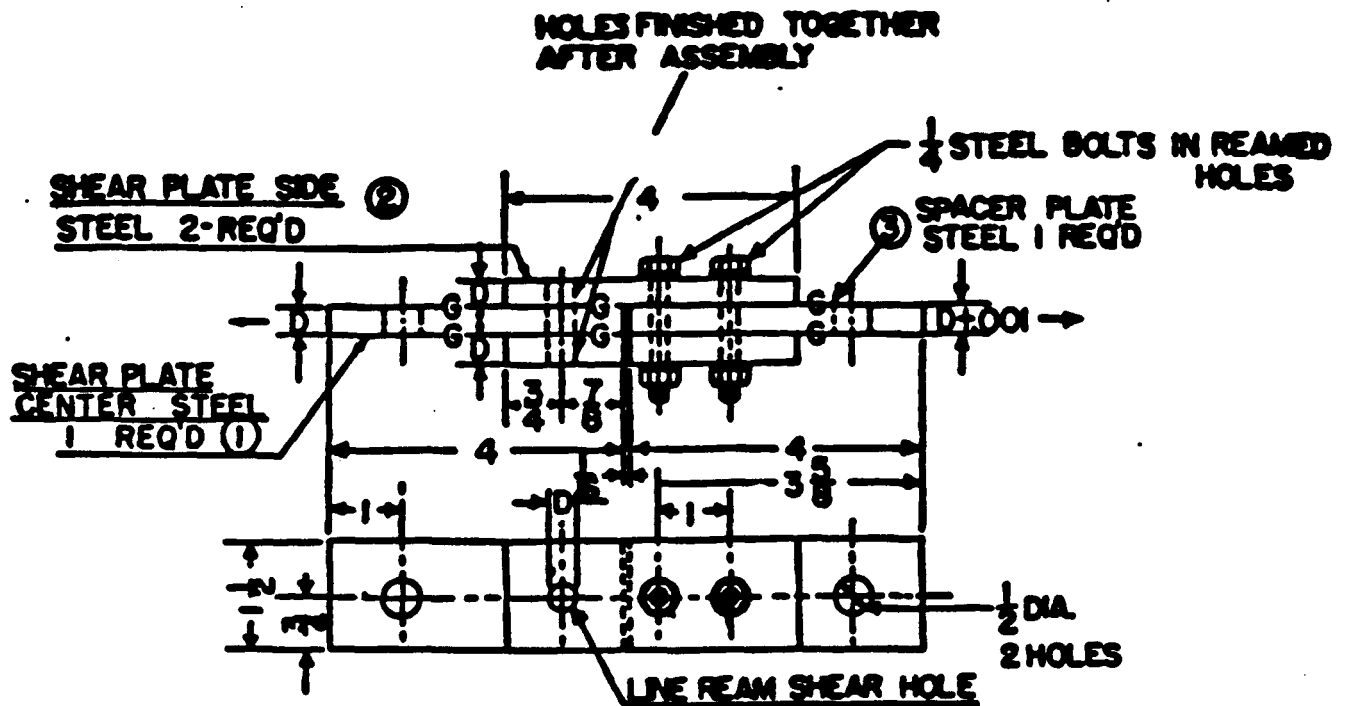
NOTE 1. - Cold rolled steel

NOTE 2. - Furnished four steel machine screws and nuts, round head, slotted, length 1-1/4 in.

NOTE 3. - Grind surfaces denoted "Gr"

Figure 4. Support Jig Details

ORIGINAL PAGE IS
OF POOR QUALITY



- NOTES:
1. D is nominal finish of test cylinder.
 2. Finish: machine after heat treatment.
 3. Grind surfaces denoted G.
 4. Spacer plate (3) to be 0.001 inch thicker than (1).
 5. Edges of shear hole must be sharp.
 6. All dimensions in inches; tolerances—fractions $\pm 1/64$; specimen diameter = $D + 0.000, -0.001$; diameter shear hole = $D - 0.000, -0.001$.
 7. Alignment of shear holes must be maintained.
 8. Material: steel, heat-treated to Rockwell C 35-40.

Figure 5. Double Shear Jig

1 February 1988

GEORGE C. MARSHALL SPACE FLIGHT CENTER
National Aeronautics and Space Administration

SPECIFICATION

SPUN PAN PHENOLIC CLOTH, 6 oz/yd², PREIMPREGNATED

1. SCOPE

- 1.1 Scope. This specification covers one type of carbon cloth reinforcement preimpregnated with a carbon filled phenolic resin.

2. APPLICABLE DOCUMENTS

- 2.1 Government Documents. The following documents of the issue in effect on date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

SPECIFICATIONS

Military

MIL-B-131	Barrier Materials, Watervapor-proof, Grease-proof, Flexible, Heat-sealable
MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification
MIL-R-9299	Resin, Phenolic, Laminating

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

- 2.2 Non-Government Documents. Unless otherwise specified, the following documents of the latest approved issues form a part of this specification to the extent specified herein.

SPECIFICATIONS

MSFC-SPEC (USP-28) Filler, Carbon Black

MSFC-SPEC (PWB-6) Reinforcement, Carbon, Spun PAN, 6 oz/yd²

MSFC-SPEC (USP-39A) Resin, Phenylaldehyde

(Application for copies should be addressed to George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, AL 35812.)

American Society for Testing and Materials (ASTM)

ASTM C 177	Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate, Standard Test Method for
ASTM C 613	Resin Content of Carbon and Graphite Pregs by Solvent Extraction, Standard Test Method for
ASTM D 638	Tensile Properties of Plastics, Standard Test Method for
ASTM D 790	Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials, Standard Test Methods for
ASTM E 831	Linear Thermal Expansion of Solid Materials by Thermodilatometry, Standard Test Method for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

- 3.1 Qualification. The material furnished under this specification shall be a product which has been tested and has passed the qualification inspection specified herein (see 4.3 and 6.4).
- 3.2 Material. The material shall be a carbon fabric reinforcement impregnated with a carbon filled phenolic resin and supplied as broadgoods. Broadgoods roll widths shall be 36 to 48 inches and shall be rolled onto the paper core with the warp side of the impregnated fabric on the outside face of the rolls (see Figure 1).

3.2.1 Resin. The resin shall conform to the requirements of MIL-R-9299, Grade A, and also to MSFC-SPEC (USP-39A).

3.2.2 Reinforcement. The reinforcement shall be a carbon fabric conforming to MSFC-SPEC (PWB-6).

3.2.3 Resin Filler. The resin filler shall be carbon powder conforming to MSFC-SPEC (USP-28).

3.3 Properties

3.3.1 Uncured Material. The properties of the uncured material shall be in accordance with Table I.

TABLE I. UNCURED MATERIAL PROPERTIES

PROPERTY	SPECIFICATION LIMITS		RETEST	PROPERTIES <u>1/</u>
	MINIMUM	MAXIMUM	MINIMUM	MAXIMUM
Cloth Content, %	43.0	57.0	----	----
Dry Resin Solids, %	30.0	40.0	----	----
Volatile Content, %	----	5.0	----	6.0
Carbon Filler Content, %	11.0	18.0	----	----
Resin Flow, %, 1000 psi	8.0	20.0	8.0	23.0
Tack, lbs.	25	----	25	---
Sodium Content, ppm	----	100	----	----

1/ Procuring activity testing for extension of material shelf life after six months from date of supplier manufacture.

3.3.2 Cured Material. The properties of the cured material shall be in accordance with Table II.

TABLE II. CURED MATERIAL PHYSICAL AND MECHANICAL PROPERTIES
(AT ROOM TEMPERATURE UNLESS OTHERWISE SPECIFIED)

PROPERTY	LIMITS	
	MINIMUM	MAXIMUM
Density, grams per cubic centimeter (g/cc)	1.520	1.600
Residual Volatiles, percent	0.0	3.0
Resin Content, percent	28.0	36.0
Compressive Strength, psi (edgewise)		
warp direction	10,000	30,000
fill direction	TBD	TBD
Interlaminar Double Shear Strength, psi	2,500	6,000
Thermal Conductivity, Btu/ft-hr-degrees F		
at 250°F with ply	TBD	TBD
across ply	TBD	TBD
Coefficient of Thermal Expansion		
in/in-degrees F x 10 exp -6		
at 400°F across ply	2.0	18.0
with ply	-5.0	5.0
Flexural Strength, psi		
warp direction	20,000	50,000
fill direction	TBD	TBD
Tensile Strength, psi		
warp direction	10,000	30,000
fill direction	TBD	TBD

3.4 Age of Material. The material shall be shipped within 3 months of manufacturing date and shall be tested and certified within 60 days of shipment.

3.5 Process of Manufacture. Any change in process or material after initial qualification testing as required by this specification shall be cause to require requalification.

- 3.6 Storage Life. The material shall have a storage life of six months after date of manufacture when stored at 50°F maximum in sealed plastic bags with a 1-unit minimum desiccant bag conforming to MIL-D-3464. After date of receipt of the broadgoods, the maximum cumulative out-of-storage time shall be 30 days at 85°F maximum. Storage life requirements shall apply until the material has been completely cured.
- 3.6.1 Storage Life Extension. For the convenience of the procuring activity, the storage life of broadgoods may be extended for three additional 6-month periods provided the material, upon retest (see 6.5), conforms to the retest requirement for resin flow, volatile content, and tack in Table I. The maximum allowable cumulative storage life of broadgoods shall not exceed 24 months from the date of manufacture of the original broadgoods lot. Each individual roll of broadgoods must be tested for storage life extension.
- 3.6.2 Conditioning for Storage Life Extension Retest. Material which fails to meet the allowable volatile, tack and resin flow retest requirements may be conditioned to extend the storage life by subjecting the rolls which failed to meet the requirements to the following:
- Place broadgoods material in a vacuum chamber supported through core(s) to prevent material damage.
 - Expose material to a vacuum of less than one inch of mercury (Hg) absolute and a temperature of 60-90°F for a period of 7-9 days.
 - After completion of the drying process, obtain samples from outside and inside of each roll and retest for volatile content resin flow and tack.
- Failure of any of the conditioned rolls to meet the retest requirements of Table I shall require the roll to be scrapped.
- 3.6.3 Out-of-Storage Time Extension - The 30-day out-of-storage time at 85°F maximum may be extended for one additional 30-day period provided that each roll and the average of three tests has met the retest requirements for volatile content, resin flow and tack specified in Table I.
- 3.6.4 Preparation and Storage of Samples Selected for Retest. Each lot of samples shall be promptly put into an individual plastic bag, sealed, properly labeled, and stored with the broadgoods it represents. Prior to testing and removal from the plastic bag, the samples shall be conditioned at 60-80°F for two hours minimum. The samples shall be tested within 72 hours after removal from plastic bags and within 14 days after removal from the broadgoods rolls.

3.7 Workmanship

- 3.7.1 Broadgoods. The material shall have no holes, tears, non-uniform weave, resin build-up, contaminants, or other defects which would render more than 5 percent of any roll unsuitable for the intended use.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the supplier may use his own or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that materials and test results conform to prescribed requirements.
- 4.2 Classification of Inspections. The inspection requirements specified herein are classified as follows:
- a. Qualification inspection (see 4.3)
 - b. Quality conformance inspection (see 4.4).
- 4.3 Qualification Inspection. Three rolls of broadgoods from the first production lot (see 4.4.1.1) shall be subjected to all examinations and tests specified in Table III. The lots subjected to qualification inspection shall be representative of the manufacturing production from the proposed production facility.

TABLE III. QUALIFICATION INSPECTION

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5

TABLE III. QUALIFICATION INSPECTION (CONT'D.)

EXAMINATIONS AND TESTS	REQUIREMENT	TEST METHOD
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Residual Volatiles	3.3.2	4.6.3.3
Resin Content	3.3.2	4.6.3.4
Compressive Strength	3.3.2	4.6.3.5
Interlaminar Shear	3.3.2	4.6.3.6
Thermal Conductivity	3.3.2	4.6.3.7
Coefficient of Thermal Expansion	3.3.2	4.6.3.8
Flexural Strength	3.3.2	4.6.3.9
Tensile Strength	3.3.2	4.6.3.10

4.4 Quality Conformance Inspection. Quality conformance inspection shall consist of all the examinations and tests specified in Table IV performed on each lot.

TABLE IV. QUALITY CONFORMANCE INSPECTION

EXAMINATIONS AND TEST	REQUIREMENT	TEST METHOD
<u>UNCURED MATERIAL</u>		
Visual Examination	3.7 & Section 5	4.6.1
Volatile Content	3.3.1	4.6.2.1
Dry Resin Solids	3.3.1	4.6.2.2
Carbon Filler Content	3.3.1	4.6.2.2
Cloth Content	3.3.1	4.6.2.2
Resin Flow	3.3.1	4.6.2.3
Tack	3.3.1	4.6.2.4
Sodium Content	3.3.1	4.6.2.5
<u>CURED MATERIAL</u>		
Density	3.3.2	4.6.3.2
Resin Content	3.3.2	4.6.3.4
Compressive Strength (warp direction only)	3.3.2	4.6.3.5

4.4.1 Sampling. Each lot of carbon cloth phenolic shall be sampled for inspection as follows:

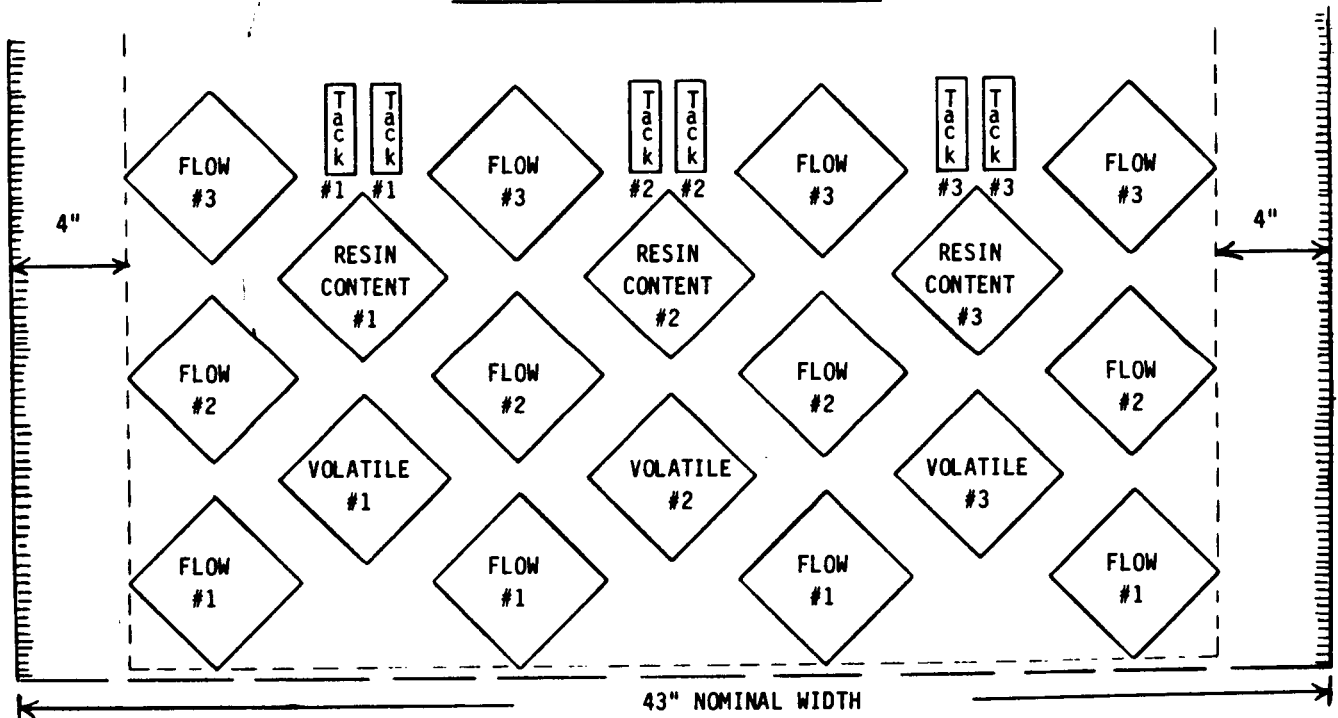
4.4.1.1 Lot. A manufacturing lot of preimpregnated material shall consist of material which is impregnated in one continuous production run, using one batch of the impregnator's resin and additives mixed in one vessel at one time.

4.4.1.1.1 Lot Size. A lot of carbon cloth phenolic shall consist of not more than 15,000 pounds.

4.4.1.2 Lot Acceptance Test Samples.

4.4.1.2.1 Sampling for Uncured Material Properties. Each sample shall be of sufficient size to perform all of the uncured material inspections and tests. Each sample shall be marked with the roll number, lot number, and the location in the roll from which the sample was taken. Each broadgoods roll in the lot shall be sampled at the exposed end of the roll, as shipped.

TYPICAL UNCURED MATERIAL SAMPLING



- 4.4.1.2.2 Sampling for Cured Material Properties. A sample of sufficient size to perform all the cured material inspections and tests specified herein shall be taken from the exposed end of each broadgoods roll selected at random from each lot. The number of rolls to be selected shall be determined as follows:

NUMBER OF ROLLS IN LOT	NUMBER OR ROLLS TO BE SELECTED FOR SAMPLING
1 to 2	All
3 to 40	3
41 to 65	4
66 to 110	5

Failure of a roll sample to conform to the requirements of this specification shall be cause to withdraw that roll and shall require that all other rolls in that lot be tested for only those material properties which failed to meet specification requirements.

- 4.5 Test Requirements. Three tests shall be performed for each of the material properties specified herein on the samples taken from each broadgoods roll sampled. The average values of the test results and at least two of the three individual test values shall be used to determine conformance to the requirements of this specification.
- 4.5.1 Test Conditions. Unless otherwise specified, all tests shall be performed under ambient conditions.
- 4.5.1.1 Supplier Retest. When the average test value of any material property fails to conform to Tables I and II and the cause of the failure can be attributed to improper preparation of the specimen or testing machine anomaly, new tests shall be conducted on a sample from the same broadgoods roll from which the failed specimens were removed.
- 4.6 Test Methods. The following test methods and procedures shall be used. Unless otherwise specified, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal. When a referenced document provides the test method description, that document applies only to the extent of specifying the method; additional requirements, such as conditioning or data reporting, imposed by the referenced document are not applicable. Wherever a value is preceded by "approximately", a tolerance of plus or minus 20 percent shall be used.

4.6.1 Visual Examination. Each roll of preimpregnated cloth shall be visually examined for conformance to 3.7 and Section 5 before supplier shipment.

4.6.1.1 Visual Examination for Conformance to Special Shipping and Marking Requirements. Temperature record charts of the ambient temperature to which the material was exposed during shipment shall be examined for conformance to 5.2. Marking shall be visually inspected for conformance to 5.3.

4.6.2 Uncured Material Tests.

4.6.2.1 Volatile Content. The volatile content of each sample of the uncured material shall be determined in accordance with the following:

- a. Cut a 16 ± 2 square inch specimen.
- b. Weigh the specimen to the nearest 0.01 gram (g) (W1).
- c. Place specimen in a recirculating oven preheated and stabilized to $325 \pm 10^\circ\text{F}$ for 10 ± 1 minutes. Specimens should be placed to get adequate air circulation.
- d. Remove the specimen and within one (1) minute, weigh to the nearest 0.01 g (W2).
- e. Calculate percent volatiles as follows:

$$\text{Percent volatiles} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g

W2 = final weight of specimen, g

4.6.2.2 Resin Solids, Filler Content, and Cloth Content. Resin solids, shall be determined for each sample in accordance with the soxhlet extraction method specified in ASTM C 613 and the following:

- a. Precondition a paper thimble at $325 \pm 5^\circ\text{F}$ for 120 minutes. Weigh immediately to .001 gm (W1) and store in a desiccator with the recorded weight until ready to use.
- b. Reweigh the thimble immediately before addition of the sample (W2) so an accurate sample weight is established. Place a bias cut 2.0 x 4.0 inch prepreg sample into the thimble and weigh to .001 gm (W3).

- c. Using the volatile content obtained in 4.6.2.1e, calculate the devolatilized sample weight by using the formula:

$$W = \text{devolatilized sample weight} = (W3 - W2) \times \left(1 - \frac{V}{100}\right)$$

Where W2 = Thimble weight immediately before sample addition, gm.

W3 = Thimble weight plus sample, gm.

V = Volatile content, wt. %

- d. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, allow the solvent to drain, and dry to constant weight at $325 \pm 5^\circ\text{F}$ for 2 hours minimum in a forced air oven. Remove from the oven in sets of three, and immediately weigh to .001 gm (W4).

- e. If filler has appeared in the extract, determine the filler weight lost by the filtering and crucible technique in ASTM C-613, and add the recovered filler weight to the dried thimble plus sample weight (W4).

- f. Calculate the dry resin content (resin solids) as follows:

$$\text{DRC, wt. \%} = 100 \left(1 - \frac{W4 - W1}{W}\right)$$

Where W = Devolatilized sample weight, gm.

W1 = Weight of dried thimble, gm.

W4 = Weight of thimble plus extracted sample, gm.

- g. Filler content can be calculated by utilizing the filler content in the resin mix before impregnation as follows:

$$\text{Filler content, wt. \%} = \frac{(\text{DRC}) (\text{FC})}{(100 - \text{FC})}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content of the resin mix

- h. Cloth content can be calculated as follows:

$$\text{Cloth content, wt. \%} = 100 - \text{DRC} - \text{FC}$$

Where DRC = % dry resin content, 4.6.2.2f

FC = % filler content 4.6.2.2g

4.6.2.2.1 As an alternative method for determining filler content and cloth content, the following procedure may be used:

- a. Follow the procedures of 4.6.2.2 thru 4.6.2.2c.
- b. Reflux as per ASTM C-613 for a minimum of 4 hours. Remove the thimble, and allow the solvent to drain.
- c. Carefully open the thimble, remove the cloth and place the cloth in the bottom of a clean 250 ml beaker. Add 20-25 ml of ethyl alcohol and gently stir to remove any remaining filler.
- d. Transfer the liquid solution back to the original thimble, removing any visible fibers and keeping the fibers with the fabric.
- e. Rinse the fabric with ethyl alcohol several times to ensure complete removal of the filler from the fabric and add the rinses back to the original thimble. Make the final rinse acetone to remove all traces of ethyl alcohol.
- f. Dry the fabric in an oven for a minimum of 30 minutes at 325°F, cool in a desiccator to room temperature and reweigh to the nearest .001 gm (W5). This is the dry cloth weight.
- g. Close the top of the thimble and dry to a constant weight at 325 ± 5°F for 2 hours minimum in a forced air oven. Remove from the oven and immediately weigh to .001 gm (W6).
- h. Calculate the resin solids, filler content and cloth content as follows:

$$\text{Filler content, wt. \%} = \left(\frac{W6 - W1}{W} \right) \times 100$$

$$\text{Cloth content, wt. \%} = \left(\frac{W5}{W} \right) \times 100$$

$$\text{Resin solids content, wt. \%} = 100 - \left(\frac{W6 + W5 - W1}{W} \right)$$

Where W = devolatilized sample weight from 4.6.2.2

W1 = weight of dried thimble, gm.

W5 = weight of dried cloth, gm.

W6 = weight of thimble plus filler, gm.

4.6.2.3 Resin Flow. Resin flow of each sample shall be determined in accordance with the following:

- a. Cut four 4-inch by 4-inch \pm 1/8-inch squares across the width of the fabric. All squares shall be bias cut to eliminate fiber loss in testing. Stack the squares uniformly on each other to make a specimen.
- b. Weigh the specimen of four plies to the nearest 0.01 g and record as W1.
- c. Place the specimen between release film. Preheat the press to $325 \pm 10^\circ\text{F}$, position the specimen in the middle of the press plate and apply the press load of 1000 ± 50 pounds per square inch gauge (psig) immediately. Press-load the specimen for a minimum of 10 minutes at $325 \pm 10^\circ\text{F}$.
- d. Remove the specimen from the press and cool to ambient temperature.
- e. Using the edge of a metal plate or knife, scrape off the resin flash to the original size of the specimen. Do not remove any reinforcement from the original dimensions.

NOTE: Include any fibers which may be displaced and scraped off during the process in the weighed back specimen weight (W2).

- f. Reweigh the specimen to the nearest 0.01 g and record as W2.
- g. Calculate the percent resin flow as follows:

$$\text{Percent flow} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = uncured weight of specimen, g
W2 = final weight of specimen, g

4.6.2.4 Tack. Tack shall be determined in accordance with the following:

- a. Cut two 1.0 x 4" strips and place them lengthwise with a 1.0" overlap (wrap to fill side) into a sheet of folded cellophane.
- b. Place the wrapped specimen onto a preheated $190 \pm 5^\circ\text{F}$ press. Close the press to 50 psi as rapidly as possible and hold this pressure for 60 seconds.

- c. Remove the specimen from the press, and place between two 1/8 inch thick room temperature plates, and allow to cool to room temperature.
- d. Using an appropriate tensile testing device, apply a constant rate of loading to obtain sample failure within 5 to 10 seconds. Record and report the average load at failure of three determinations.

4.6.2.5 Sodium Content. Sodium content shall be determined in accordance with the following:

NOTE: Care should be exercised not to contaminate the sample with sodium. Samples should not be touched with bare hands.

- a. Take specimens which weigh approximately 3 g. Place test specimens in individual beakers and dry in an air circulating oven at $225 \pm 5^{\circ}\text{F}$ for a minimum of one hour.
- b. Remove the specimens from the oven and cool in a desiccator.
- c. Weigh approximately 2 g of each cooled specimen to the nearest 0.1 milligram (mg) into separate tared ceramic or platinum crucibles.
- d. Heat the crucibles in a muffle furnace at $600 \pm 15^{\circ}\text{C}$ for 16-18 hours or until constant weight is achieved.
- e. Remove the crucibles containing the ashed specimens, place in a desiccator and cool.
- f. Wash down the walls of each crucible with distilled water and add approximately 5 ml concentrated hydrochloric acid to each.
- g. Bring the acid mixture to a boil, cool the crucibles, transfer the contents of each to a 1 liter volumetric flask and dilute to volume with distilled water.
- h. Determine the sodium content of each flask by flame emission at 589 nanometers (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

4.6.3 Cured Material Tests.

4.6.3.1 Preparation of Test Panels. Cured material tests shall be determined on test panels prepared in accordance with the following, when referenced:

- a. Cut sufficient plies to make the specified panel thickness and minimum size required, maintaining and marking uniform orientation of cloth fiber direction.
- b. Wrap the stack of plies $1\frac{1}{2}$ times with a suitable release film such that the upper and lower surfaces and two sides are completely encased by the release film. The open ends allow for degassing and resin flow.
- c. Center the wrapped plies between two $\frac{1}{8}$ " thick smooth steel caul plates and center the caul plates in a press preheated to $325 \pm 10^\circ\text{F}$, starting the cure timer.
- d. Apply contact pressure (nominal 50-lb. load) and maintain for $30 \pm$ seconds. Increase the load rapidly to approximately 200-lb. load and immediately release the pressure until a clearance can be observed above the top caul plate.
- e. Immediately reapply contact pressure and "bump" the laminate as indicated above for one additional cycle.
- f. Increase the pressure slowly (over approximately 4 minutes) to 1000 ± 50 psi, controlling the rate of pressure application to minimize resin flow at the laminate edge.
- g. Maintain the pressure and temperature for 120 ± 15 minutes. Turn-off the platen heaters and allow the laminate to cool to below 150°F under pressure. Remove from the press, remove release film and resin flash. Do not use the outer $\frac{1}{2}$ " of the test panel for preparation of specimens, unless specifically directed.

4.6.3.2 Density. Density shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches by 5 inches minimum, prepared as per 4.6.3.1.
- b. Cut a specimen 1-inch by 1-inch by panel thickness at random from the test panel. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry 20 minutes, minimum, before testing.
- c. Weigh and record the weight of the specimen in air to the nearest 0.1 mg (W1).

d. Weigh the specimen in water at $73 \pm 3^\circ\text{F}$ and record the weight to the nearest 0.1 mg using a wire and sinker if required (W2).

e. Calculate density using water as follows:

$$\text{Density (g/cc)} = \frac{0.9975 W1}{(W1 + W3 - W2)}$$

Where: W1 = weight of specimen in air

W2 = weight of specimen, wire, and sinker suspended in water

W3 = weight of wire and sinker suspended in water to the same depth as in the W2 determination to the nearest 0.1 mg.

4.6.3.3 Residual Volatile Content. Residual volatile content shall be determined in accordance with the following:

a. Cut sufficient plies to produce a test panel 0.250 ± 0.050 inches thick and 4 inches by 4 inches, prepared as per 4.6.3.1.

b. Cut a specimen 1.0 inch by 1.0 inch by panel thickness from the test panel; do not machine the top and bottom molded surfaces. Wipe the specimen clean using MEK and allow to air dry 20 minutes minimum.

c. Place the specimen in a desiccator and desiccate for 18 hours minimum.

d. Weigh the specimen to the nearest 0.01 gram and record as W1.

e. Place the specimen in an air circulating oven, preheated and stabilized at $325 \pm 10^\circ\text{F}$. Maintain the specimen at a temperature of $325 \pm 10^\circ\text{F}$ for 4 hours.

f. Remove the specimen from the oven and cool in a desiccator for approximately 30 minutes or until the specimen reaches room temperature.

g. Reweigh the specimen to the nearest 0.01 gram. Record as W2.

h. Calculate the percent residual volatiles as follows:

$$\text{Residual volatile, \%} = \left(\frac{W1 - W2}{W1} \right) \times 100$$

Where: W1 = original weight of specimen, g

W2 = devolatilized weight of specimen, g

4.6.3.4 Resin Content. Resin content shall be determined in accordance with the following:

a. Prepare test specimens as follows:

- (1) Cut 32 rectangles (1/2 inch by 5 inches nominal) from a prepreg sample.
- (2) Stage (see 6.3.1) the 32 rectangles at $255 \pm 5^{\circ}\text{F}$ for approximately 20 minutes to reduce flow.
- (3) Using the 32 rectangles, mold a bar 1/2 inch by 1/2 inch by 5 inches.
- (4) Cure the specimen at $325 \pm 10^{\circ}\text{F}$ and 1000 ± 50 psi for 10 ± 1 minutes.

NOTE: If there is any resin flash, repeat and use a longer staging time.

- (5) Cut the bar into approximately three equal lengths to form three test specimens.

b. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry for 20 minutes, minimum, before testing.

c. Record specimen weight to the nearest 0.001 g (W).

d. Place the test specimen in the Vycor test tube and tightly attach the tube to the test apparatus (see Figure 2).

e. Start the vacuum pump and evacuate the system to less than two inches of Hg absolute. Check for air leaks by clamping off the hose to the vacuum pump and noting the manometer. The leak rate must be less than 0.20 inch of Hg per minute. Recheck joints until this level is achieved.

f. With the vacuum on and the pressure stabilized, place the Fisher burner under the specimen. Adjust flame area to completely cover the sample.

NOTE: A tube furnace maintained at $1500 \pm 25^{\circ}\text{F}$ may be used in place of the Fisher burner.

g. Continue pyrolysis for approximately 30 minutes or until the manometer or gauge reading is the same reading as it was prior to pyrolysis, whichever is longer.

NOTE: If residue collects in the Vycor test tube, place the Fisher burners under those areas until the burnout is complete.

- h. When pyrolysis is complete, turn off burners and cool under vacuum until specimen is at room temperature.
- i. When the specimen is cool, turn off vacuum and carefully let air into the system. When pressure equilibrium is reached, remove specimen from test tube and weigh to the nearest 0.001 g (W1).
- j. Calculate dry resin content as follows:

$$\text{Dry resin content, percent} = \left(1.00 - \frac{W1}{W}\right) \times 100 \times K$$

Where: W1 = weight of specimen after pyrolysis, g

W2 = weight of specimen prior to pyrolysis, g

K = a constant as shown in Table V

TABLE V. K FACTORS

U.S. POLYMERIC FM 5839
TBD

4.6.3.5 Compressive Strength. Compressive strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 0.125 ± 0.025 inches thick and 4 inches minimum by 5 inches minimum, prepared as per 4.6.3.1.
- b. Machine a specimen to the dimensions shown in Figure 3.
- c. Measure the width and thickness of the specimen to the nearest 0.001 inch in the gauge section area. Calculate and record the cross-sectional area.
- d. Place the specimen in the support jig (see Figure 4), centered so the ends project an even amount beyond both ends of the jig. Tighten finger tight only.

- e. Place the specimen and the support jig in the testing machine and align so that the specimen ends are parallel with the surface of the compression tool. Adjust the crosshead of the testing machine until it just contacts the top of the specimen.
- f. Set the speed control at an indicated crosshead rate of 0.050 inch per minute and start the machine.
- g. Record the maximum load carried by the specimen during the test.
- h. Calculate the compressive strength by dividing the maximum load by the original cross-section area.

4.6.3.6 Interlaminar Shear. Interlaminar shear strength shall be determined in accordance with the following:

- a. Cut sufficient plies to produce a test panel 1.5 ± 0.25 inch thick across the plies by minimum dimensions of 4.0 by 4.0 inches.
- b. Cure at 1000 ± 50 psi and $325 \pm 10^\circ\text{F}$ for 120 ± 15 minutes.
- c. After cure, remove the panel from the press and allow the panel to cool to room temperature.
- d. Cut a specimen of sufficient size to produce a 0.375 inch diameter by 1.125 inch minimum length finished specimen with the length being perpendicular to the plies from the test panel.

NOTE: Do not use the outer 1/2 inch of the test panel for specimen preparation.

- e. Center the specimen to get the length of the cylindrical specimen perpendicular to the plies and machine the specimens to a diameter of $0.375 +0.000, -0.002$ inch diameter.
- f. Measure the diameter of the test specimen and calculate the cross-sectional area (A).
- g. Place the specimen in the 0.375 inch diameter three plate double-shear jig shown in Figure 5.
- h. Place the shear fixture in the testing machine, align, and pin the ends of the fixture.
- i. Set the machine at an indicated crosshead speed of 0.015 to 0.025 inch per minute and test.

- j. Record the maximum load carried by the specimen during the test and record as L.
- k. Calculations:

$$\text{Interlaminar shear (psi)} = \frac{L}{2A}$$

Where: L = the maximum load carried by the specimen during testing in pounds

A = the original cross-sectional area of the specimen in square inches.

4.6.3.7 Thermal Conductivity. Thermal conductivity shall be determined in accordance with ASTM C 177 or equivalent and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Sample thickness is dependent on the dimensions of the heating unit and shall be restricted to limit the theoretical error to 1%.
- c. Determine both cross ply and with ply thermal conductivity at 250°F. Faces of the specimen must be parallel to 1% of specimen thickness.

4.6.3.8 Coefficient of Thermal Expansion. The coefficient of thermal expansion (see 6.3.2) shall be determined in accordance with ASTM E 831 and the following:

- a. Prepare a test panel of required size as per 4.6.3.1. Existing panels may be utilized as appropriate.
- b. Determine the coefficient of thermal expansion in both the with ply or parallel, and cross ply or perpendicular direction of the specimens.

4.6.3.9 Flexural Strength. Flexural strength shall be determined in accordance with ASTM D 790 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each of the desired yarn orientations, prepared as per 4.6.3.1.
- b. Test per method I, procedure A with a span-to-depth ratio of 16 to 1 with three specimens for each sample.

4.6.3.10 Tensile Strength. Tensile strength shall be determined in accordance with ASTM D 638 and the following:

- a. Cut sufficient plies to produce a test panel $0.125 \pm .025$ inches thick and of sufficient size to obtain a minimum of three specimens in each desired yarn orientations, as per 4.6.3.1.
- b. Test with Type I specimens, with dimensions and tolerances for material thickness 0.28 or under, and testing speed of 0.2 in/min.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. The rolls shall be in sealed bags conforming to MIL-B-131, Type 1, Class 1 barrier material. Each broadgoods roll shall be separately wrapped with a 1-unit, minimum, dessicant bag conforming to MIL-D-3464 enclosed within each bag.

5.1.1 Broadgoods Shipping Container. The broadgoods shall be shipped in a container that can be opened from the top and will suspend both ends of the roll in the container.

5.2 Special Shipping Requirements. During shipment, the material temperature shall be maintained at or below 90°F. The ambient temperature to which the material is exposed during shipment shall be recorded with a temperature recording device provided by the procuring activity and placed with each lot shipment by the supplier.

5.3 Marking. Unless otherwise specified in the purchase document, each container and roll shall be marked for identification and shipment and shall include the following:

- a. Supplier lot number and material name
- b. Purchase document number
- c. Acceptance stock and lot number
- d. Number of rolls
- e. Roll net weight
- f. Container identification number
- g. Date of manufacture
- h. Mark each roll on inside of spool with supplier name, roll number, lot number and material name with ink stamp, stencil or adhesive label.

- i. This specification number and revision letter
- j. Mark each container on two opposite sides with the following:

**TEMPERATURE SENSITIVE MATERIAL
DO NOT EXPOSE TO TEMPERATURES EXCEEDING 90°F**

6. NOTES

- 6.1 Intended Use. The carbon cloth phenolic covered by this specification is intended for use in the manufacture of rocket motor nozzle components.
- 6.2 Ordering Data. Purchase documents should specify the following:
 - a. Title, number, and date of this specification
 - b. Special marking, packing, packaging, and shipping
 - c. Data retention requirements
 - d. Broadgoods roll width
 - e. Qualification sampling requirements (if required)
 - f. Number of copies of inspection and test data
 - g. Type of material: broadgoods or trimmed broadgoods
 - h. Acceptance stock and lot number
- 6.3 Definitions.
 - 6.3.1 Stage. A method of stabilizing and retaining the phenolic polymer in a solid state on the carbon cloth by temperature cycling.
 - 6.3.2 Coefficient of Thermal Expansion. The slope of the straight line intersecting the linear expansion curve at 75°F and 400°F expressed as change in length per unit length per °F.
- 6.4 Qualification. The procuring activity reserves the right to purchase only those products which have been previously tested and found to meet the requirements of this specification and which have proved satisfactory in one full scale nozzle static test. Prospective suppliers whose products have not been tested and found satisfactory are required to have their products tested in order that they may be considered eligible to be awarded contracts for the product covered by this specification.
- 6.5 Storage. After receipt of the material, the procuring activity is responsible for storage and for retesting to determine storage life extension.

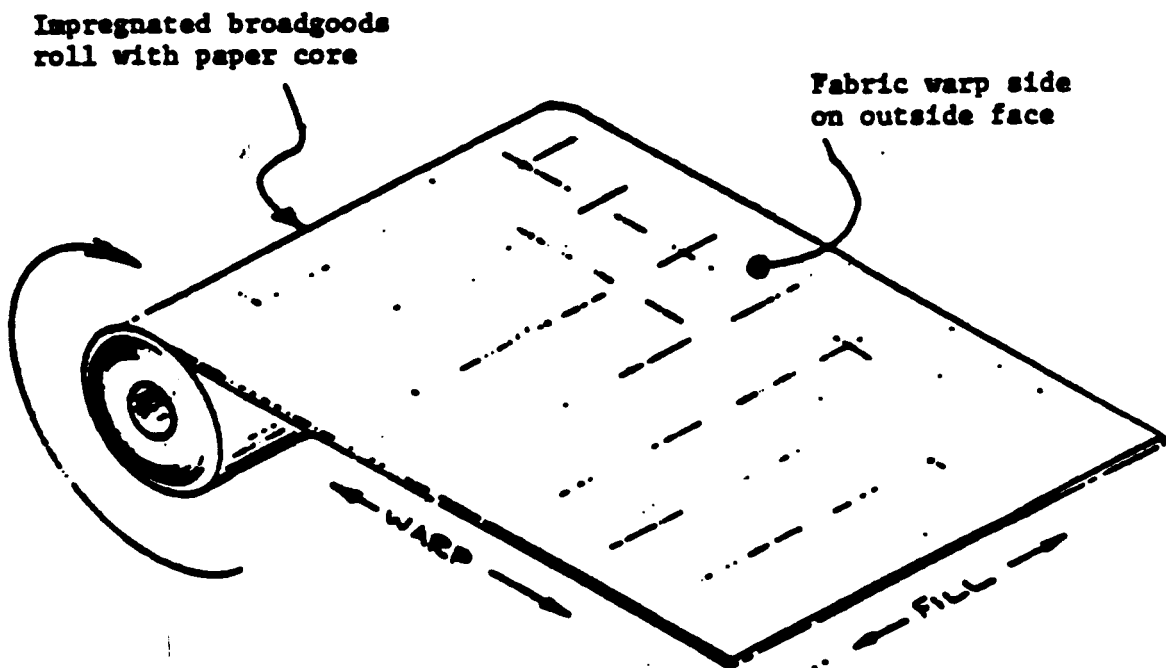


Figure 1. Requirements for Broadgoods Rolls

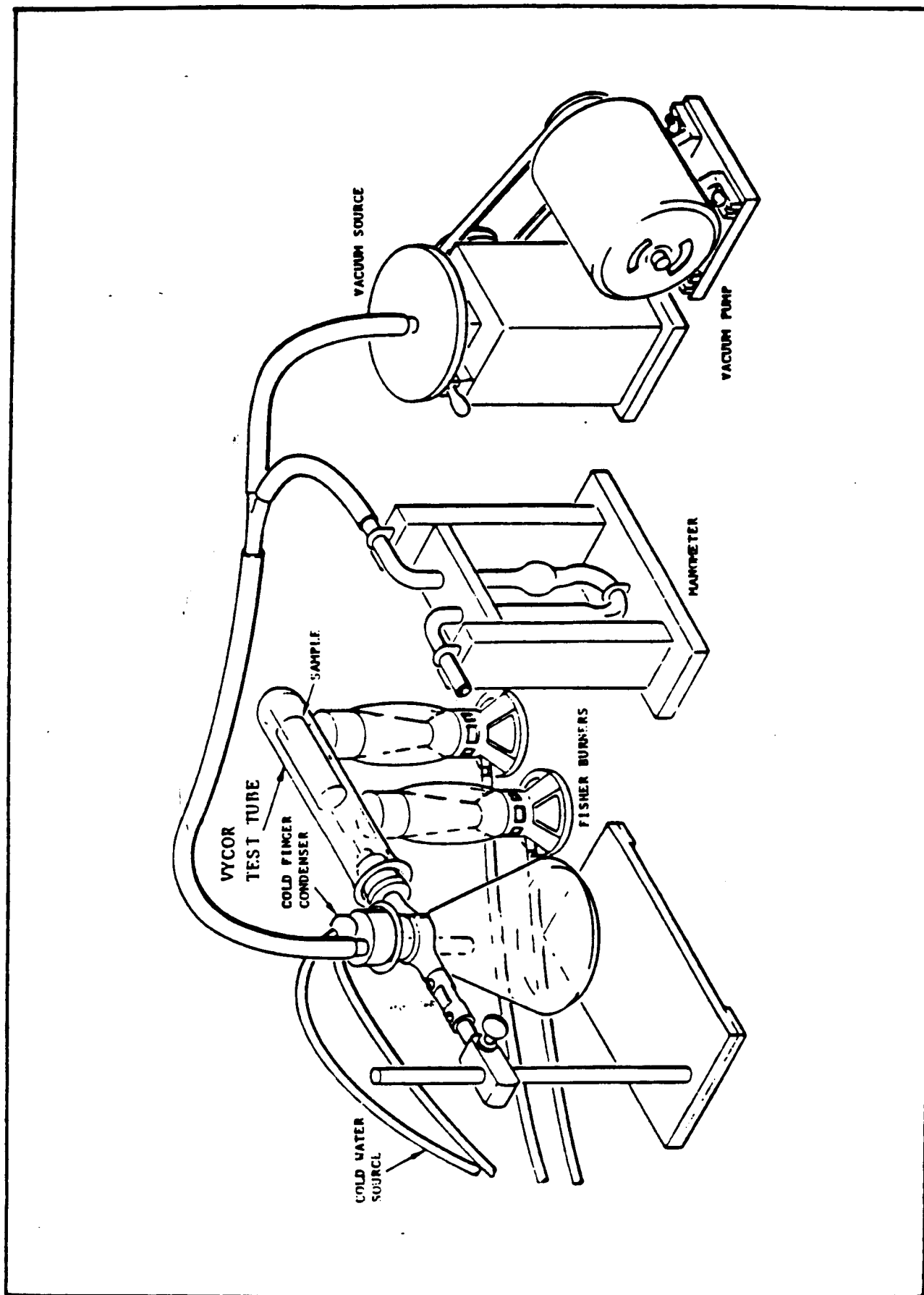
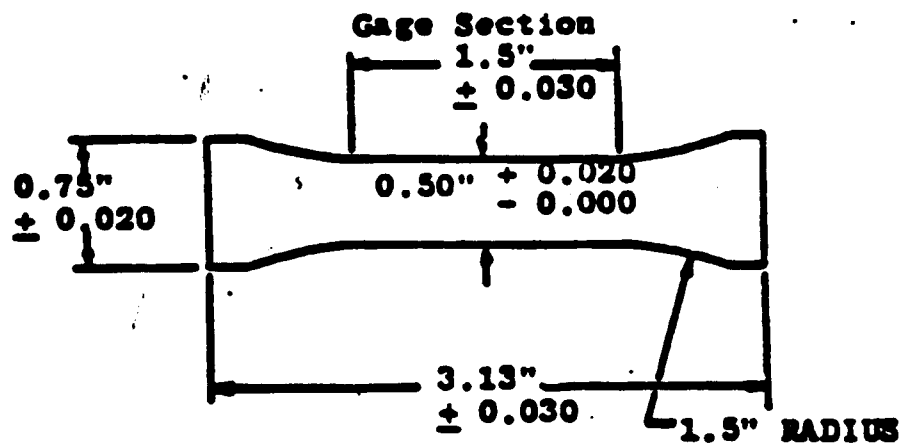


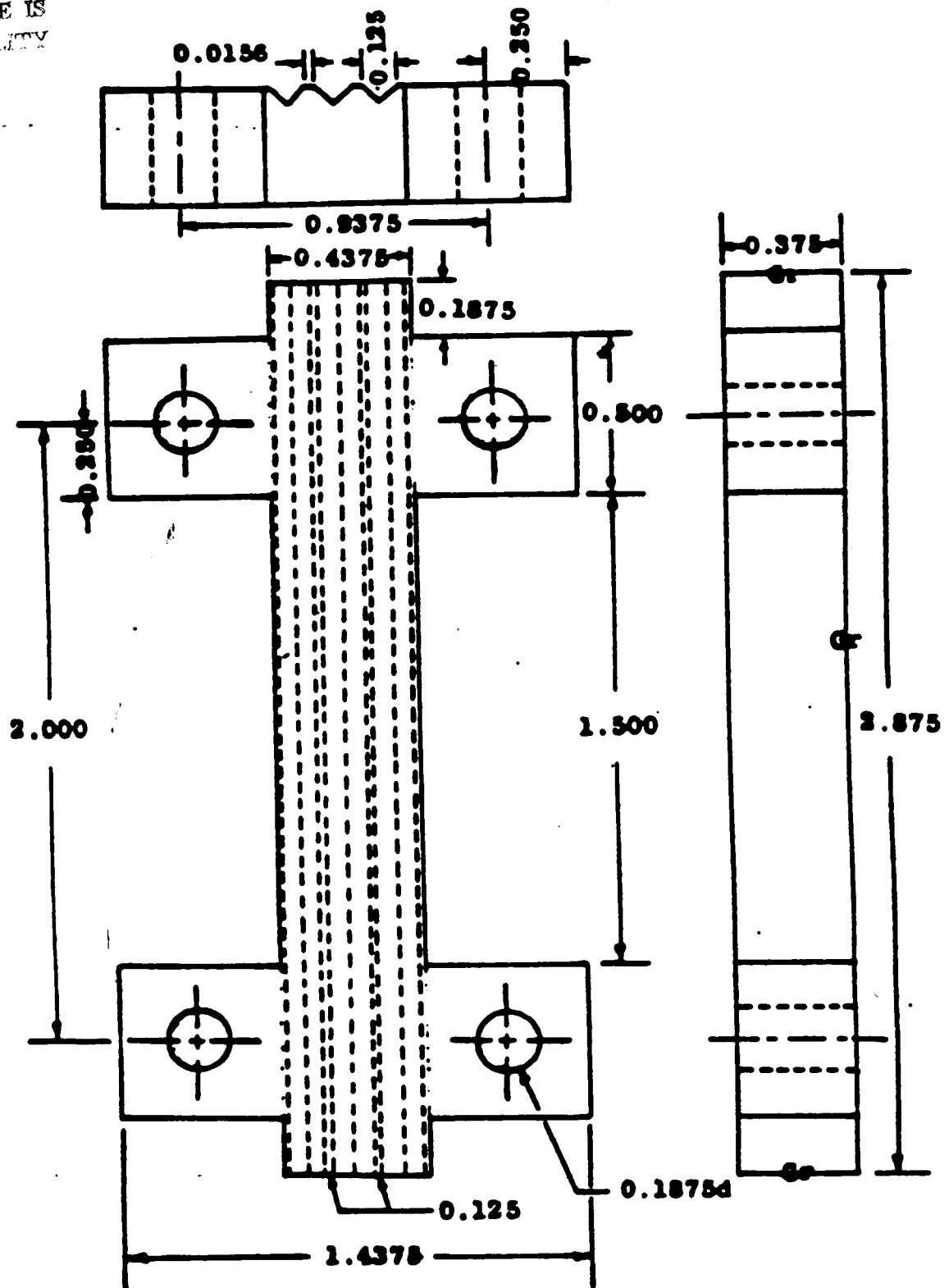
Figure 2. Resin Content Apparatus Setup



Test specimen thickness = 0.125 ± 0.025 inch

Figure 3. Compressive Strength Test Specimen

ORIGINAL PAGE IS
OF POOR QUALITY



NOTE 1. - Cold rolled steel

NOTE 2. - Furnished four steel machine screws and nuts, round head, slotted, length 1-1/4 in.

NOTE 3. - Grind surfaces denoted "Gr"

Figure 4. Support Jig Details

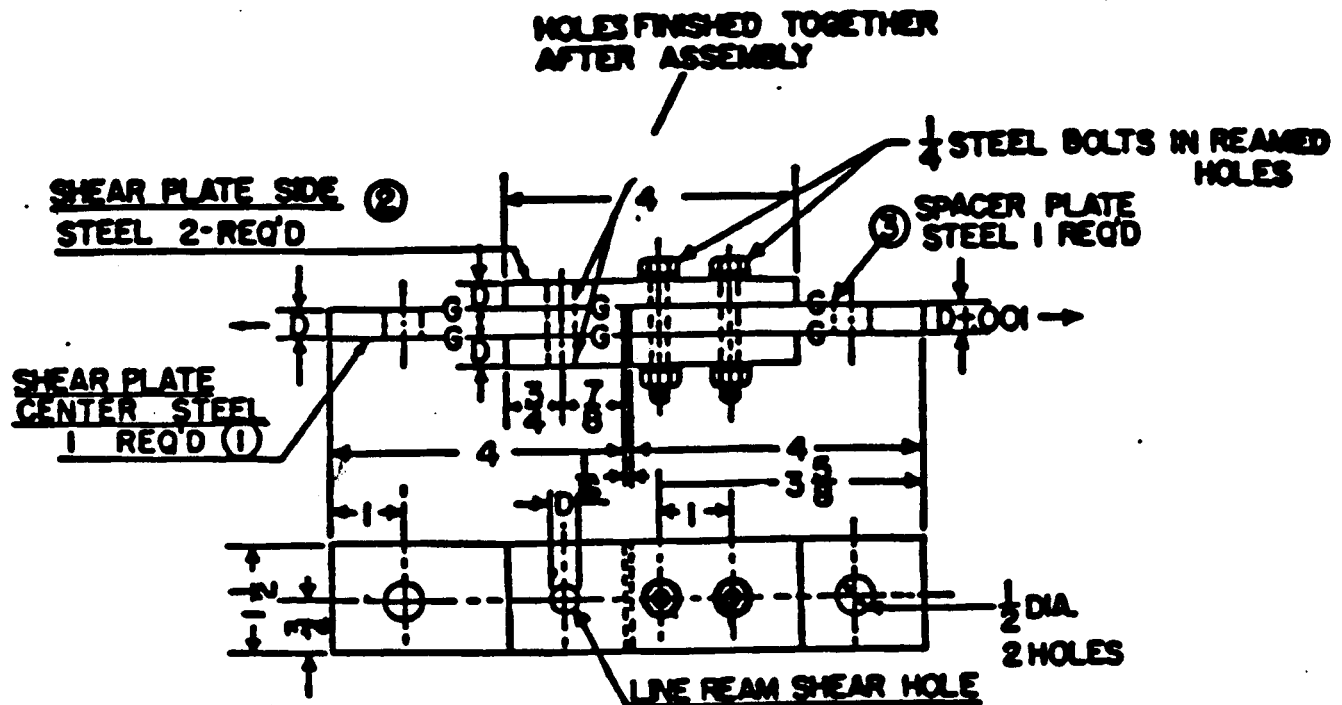


TABLE OF CONTENTS
CHEMICAL TEST METHODS

NAS8-36298

U.S. POLYMERIC O.E. 71108

<u>Number</u>	<u>Subject</u>
CTM-1B	pH of Resins and Aqueous Solutions
CTM-5B	Degree of Core using Chang's Index
CTM-6D	Resin Content by Soxhlet Extraction
CTM-14B	Resin Content of Prepreg Material by Pyrolysis
CTM-18A	Degree of Polymerization by Acetone Extraction
CTM-21C	Degree of Advancement of Phenolic Resins by Infrared Spectrophotometry
CTM-24B	pH of Raw Fabrics
CTM-24B	Amplifying Remarks
CTM-49A	Liquid Chromatography & Gel Permeation of Phenolic Resins
CTM-49A	Amplifying Remarks
CTM-50A	Differential Scanning Calorimetry
CTM-50A	Amplifying Remarks
CTM-51	Thermogravimetric Analysis
Appendix A	CCA-3 Fabric (512°C Test)
Appendix B	CCA-3 Fabric (1000°C Test)
CTM-51	Amplifying Remarks
CTM-53B	Atomic Absorption Determination of Alkaline Impurities
CTM-53B	Amplifying Remarks
CTM-55	Quality Control of Solvents Using Gas Chromatography
Addendum A	Solvent Identification in Phenolics
Addendum B	Phenol by Gas Chromatography
CTM-57A	Rheometric Dynamic Spectrometry
CTM-72	Particle Size by the Horiba Analyzer

NOTE: Individual pages are not numbered except in sequence
for each test method.

SUBJECT:

CHEMICAL TEST METHOD FOR:
PH OF RESINS AND AQUEOUS SOLUTIONS

APPROVED

PREPARED BY: * P. H. Davis 7-14-66

APPROVAL SIGNATURES:

GENERAL MANAGER DATE N/A	ENGINEERING DATE N/A	PRODUCTION DATE N/A	PRODUCTION CONTROL DATE N/A
PURCHASING DATE N/A	SALES DATE N/A		QUALITY ASSURANCE* H.M. Toellner DATE 15 July 1966

1. SCOPE

- 1.1 Scope. This method describes a procedure for the determination of pH of aqueous solutions and liquid resins.
- 1.2 Equivalent method. This method is similar to ASTM-E70-52T and will yield equivalent results.

2. EQUIPMENT AND MATERIALS

- 2.1 Equipment.
 - (a) pH meter with glass electrodes
 - (b) Beakers, 200 ml., tall form
- 2.2 Materials.
 - (a) Buffer standards of pH 4, 7, and 10
 - (b) Distilled water
 - (c) Denatured ethyl alcohol

3. SAMPLING

- 3.1 Sample size. A minimum sample of 100 ml. is required to immerse the electrodes.
- 3.2 Number of determinations. Unless otherwise specified, duplicate determinations will be run on each sample.

4. PROCEDURE

- 4.1 Standardization of pH meter. The pH meter must be standardized daily prior to use as follows (see 6.1):
 - (a) Allow sufficient time for complete warm-up of pH meter amplifier.
 - (b) Immerse clean electrodes in standard buffer solution.
 - (c) If necessary, set temperature control knob to temperature of buffer.
 - (d) Read pH meter
 - (e) Using the calibrating control knob, set pH meter to indicate pH value of buffer.
 - (f) Place pH meter in "Stand-by" condition.
 - (g) Remove buffer and rinse electrodes with distilled water.

SUBJECT:

CHEMICAL TEST METHOD FOR:
pH OF RESINS AND AQUEOUS SOLUTIONS

APPROVED

P.H. Davis
PREPARED BY: P. H. Davis 7-14-66

4.2 Determination of pH. The pH of the solution to be tested will be as follows:

- (a) Immerse electrodes in sample.
- (b) If necessary, set temperature control knob to temperature of samples.
- (c) Read pH meter.
- (d) Remove sample and rinse electrodes thoroughly with proper solvent.

5. CALCULATIONS AND REPORTING

5.1 Reporting. If the range of determinations exceeds 0.1 pH units, the results should be considered suspect. Report the average of duplicate determinations to two significant figures.

6. NOTES

6.1 Standardization. The pH meter should be calibrated weekly in accordance with SCM-7-02. If for any reason the pH meter cannot be calibrated properly, consult SCM-7-02 for possible electrode failure and electrode servicing.



QUALITY ASSURANCE
TEST METHODS

DATE

9/8/65

NUMBER CTM-5B

Page 1 of 3

SUBJECT:

B

CHEMICAL TEST METHOD FOR:
DEGREE OF CURE USING CHANG'S
INDEX

PREPARED BY: •

P. D. McCart

PD McCart

APPROVAL SIGNATURES:

GENERAL MANAGER	ENGINEERING	PRODUCTION	PRODUCTION CONTROL
DATE	DATE	DATE	DATE
PURCHASING	SALES	R. & D.	QUALITY ASSURANCE*
DATE	DATE	DATE <i>Sept. 20, 1965</i>	DATE <i>Sept. 16, 1965</i>

1. SCOPE

- 1.1 This method describes a procedure for an arbitrary measure of the relative degree of cure, advancement, or polymerization of liquid phenolic resins or phenolic prepreps.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Buret, 50 ml.
- (b) Balance, analytical
- (c) Triple beam balance
- (d) Pipet, 25 ml.
- (e) Aluminum cups, 1.5 gm., diameter 2-3/8 inches, height 5/8 inch
- (f) Hypodermic syringes, 2 ml., 10 ml., 30 ml. with delivery needles
- (g) Oven, forced air
- (h) Desiccator
- (i) Filtering crucibles, fritted glass, medium or fine

2.2 Materials.

- (a) Distilled water
- (b) Acetone
- (c) Filter paper, Whatman #41, #42, or equivalent

3. SAMPLING

3.1 Sample size.

3.1.1 Liquid resins.

- (a) 10 grams for resins between 50% and 100% solids
- (b) 20 grams for resins between 30% and 50% solids
- (c) For resins near 20% solids, measure solids per 4.2 and proceed to 4.3.

3.1.2 Prepreg materials.

- (a) 15 grams for materials of between 40% and 50% dry resin content
- (b) 20 grams for materials of between 30% and 40% dry resin content

*Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY ASSURANCE
TEST METHODS

DATE

9/8/65

NUMBER CTM-5B

Page 2 of 3

SUBJECT:

B

CHEMICAL TEST METHOD FOR:
DEGREE OF CURE USING CHANG'S
INDEX

PREPARED BY: *

P. D. McCart

P. D. McCart

4. PROCEDURE

4.1 Preparation of intermediate solutions. Prepare duplicate solutions as follows:

- 4.1.1 Liquid resin. Mix in a flask the appropriate sample size, weighed on the triple beam balance, with about 50 ml. of acetone. Determine the resin solids of the mixture according to the method of 4.2.
- 4.1.2 Prepreg materials. Soak the appropriate sample size, chopped to about 1/2" x 1/2" and weighed in a flask on a triple beam balance, with 50 ml. of acetone for 15 minutes minimum. Swirl occasionally. Filter the extracted material through Whatman #41 or #42 filter paper. If the material contains fine filler, thorough filtration may require the use of a medium or fine fritted glass filtration crucible. Determine the resin solids of the solution per 4.2.

4.2 Determination of resin solids of intermediate solutions. Determine resin solids of intermediate solutions as follows:

- 4.2.1 Weighing and devolatilization. Weigh on the analytical balance by difference from a 2 ml. syringe, 2 ml. of intermediate solution [or liquid resin of 3.1.1(c)]. Discharge the solution into a tared aluminum solids dish. Devolatilize in a forced air oven at $275 \pm 5^{\circ}\text{F}$ for 30 minutes. Cool in a desiccator and reweigh.
- 4.2.2 Calculation of resin solids. Calculate resin solids as follows:
- $$\text{Resin solids, wt. \%} = \frac{\text{Weight of sample after devolatilization}}{\text{Weight of sample before devolatilization}} \times 100$$

4.3 Preparation of 4.0% resin solids solution. Prepare the final solution as follows:

- 4.3.1 Calculations for dilution. Calculate W_1 , the weight in grams of intermediate solution to be used as follows:

$$W_1 = \frac{160}{\% \text{ resin solids from 4.2.2}}$$

Calculate W_2 , the weight of acetone to be used, as follows:

$$W_2 = 40 - W_1$$

- 4.3.2 Dilution. On the analytical balance, introduce W_1 into a 125 ml. flask from a large syringe as quickly as possible to the nearest 0.001 gm. Immediately introduce W_2 into the flask from a large syringe as quickly as possible. Long, steel delivery needles simplify these operations. Stopper the flask and swirl lightly to mix.
- 4.3.3 Verification of 4.0% resin solids. For adequate precision, solids must be between 3.95% and 4.0%. Occasionally verify resin solids by the procedure of 4.2.2 except use an 8 ml. sample size. This change in sample size keeps the total weight of resin used roughly equal for both determinations.



QUALITY ASSURANCE
TEST METHODS

DATE

9/8/65

NUMBER CTM-5B

Page 3 of 3

SUBJECT:



CHEMICAL TEST METHOD FOR:
DEGREE OF CURE USING CHANG'S
INDEX

PREPARED BY: *

P. D. McCart

4.4 Titration. Pipet 25 ml. of the final solution of 4.3 into a 125 ml. erlenmeyer flask. Titrate with distilled water to the first perceptible, persistent end point. Recognition of the end point is made easier if a strong compact light beam is shined through the flask during titration. Colloidal particles scatter the beam at the end point.

5. CALCULATIONS AND REPORTING

5.1 Reporting. Report the average number of ml. of water used as Chang's Index. Duplicate determinations should be considered suspect if they differ by more than 0.3 ml.

 P.O.S. Chemicals, Inc.	QUALITY ASSURANCE TEST METHODS	DATE 8/8/66	NUMBER CTM-6D Page 1 of 4

SUBJECT: CHEMICAL TEST METHOD FOR: RESIN CONTENT BY SOXHLET EXTRACTION	APPROVED PREPARED BY: * <i>P. D. McCart</i> 8/22/66
---	---

APPROVAL SIGNATURES:			
GENERAL MANAGER DATE N/A	ENGINEERING DATE N/A	PRODUCTION DATE N/A	PRODUCTION CONTROL DATE N/A
PURCHASING DATE N/A	SALES DATE N/A		QUALITY ASSURANCE* <i>H. M. Toellner</i> DATE 1 Sept. 1966

1. SCOPE

- 1.1 This method describes a procedure for the determination of the resin content on carbon, graphite, fortisan and nylon prepregs by soxhlet extraction. Special provisions are included to determine the resin content of filled systems.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Balance, analytical
- (b) Bottle, wash
- (c) Crucible, filtering, fritted glass, medium grade, 30 ml.
- (d) Crucible holder
- (e) Desiccator
- (f) Extraction thimbles, pyrex, extra coarse, 25 x 85 mm.
- (g) Flask, filtering, 500 ml.
- (h) Furnace, muffle
- (i) Oven, forced air (325 + 5°F)
- (j) Soxhlet extraction assembly, 125 ml. capacity with hot plate
- (k) Vacuum system

2.2 Materials.

- (a) Extraction thimbles, paper, fat-extracted, 25 x 85 mm.
- (b) Solvent, technical grade (see 4.5)
- (c) Filter, glass fiber, 5.5 cm., Reeve Angel grade 934AH or equivalent

3. SAMPLING

3.1 Sampling.

- 3.1.1 **Broadgoods.** The sample shall include the entire width of the unit being tested, less the first two inches in from each selvage edge. A sufficient number of strips, 1/2 inch by the usable width, shall be cut into 1/2 inch by 1/2 inch squares, to provide enough sample for the required number of soxhlet extractions per sample. These squares shall then be well mixed, using extreme care not to knock off any resin, before the actual samples are taken.

SUBJECT: **CHEMICAL TEST METHOD FOR:
RESIN CONTENT BY SOXHLET EXTRACTION**

APPROVED

PREPARED BY: *P. D. McCart* *8/22/66*

3.1.2 Chopped stock. Obtain a representative bulk sample of 10-15 grams and mix thoroughly, using extreme care not to knock off any resin.

3.2 Number of determinations. Unless otherwise specified, run duplicate determinations on each sample.

4. PROCEDURE

4.1 Volatile content. Determine volatile content of the prepreg per PTM-17, PTM-32, or as specified.

4.2 Thimble type. Unless otherwise specified, the following thimble types shall be used:

MATERIAL CARRIER

Carbon and graphite
Fortisan
Nylon

THIMBLE TYPE

Paper
Glass
Glass

4.3 Thimble conditioning. Unless otherwise specified, condition the thimbles as follows:

4.3.1 Paper. Identify with a soft lead pencil and dry for 2 hours at $325^{\circ} \pm 5^{\circ}\text{F}$ in a forced air oven. Remove in sets of 3 and weigh immediately to 0.001 gm. (W_1). Store in a desiccator together with recorded weight.

4.3.2 Glass. Dry for 30 minutes at $950 \pm 25^{\circ}\text{F}$ in a muffle furnace. Cool in a desiccator and weigh to 0.001 gm. (W_1).

4.4 Sample weighing. Unless otherwise specified, transfer a 2.5 ± 0.5 gm. sample to the weighed thimble and weigh to 0.001 gm. (W_3). When paper thimbles are used, they must be reweighed to 0.001 gm. (W_2) before addition of the sample. See note 6.1 for precautions regarding paper thimbles.

4.5 Extraction. Position the thimble within the soxhlet extraction assembly. Add 90 ml. of solvent, adding a portion of the 90 ml. to the extraction tube so that about 2/3 of the thimble is covered. The following solvents are suggested for the listed resins. However, experience shall dictate solvent used (see note 6.2).

RESIN

Phenolic
Epoxy
Silicone
Polyester

SUGGESTED SOLVENT

Ethyl alcohol or dimethylformamide (DMF)
Acetone or methyl ethyl ketone (MEK)
Piperidine or toluene
Acetone or MEK

Turn on the cooling water for the condenser. Turn on the hot plate to effect heating of the solvent. When condensation of the solvent occurs, adjust hot plate temperature to effect 3 to 10 reflux changes per hour. For high boiling solvents, such as DMF, it may be necessary to wrap the extraction tube with aluminum foil. Ensure that the solvent is condensing and dropping into the extraction tube. Continue to reflux for a minimum of four hours.

SUBJECT:

CHEMICAL TEST METHOD FOR:
RESIN CONTENT BY SOXHLET EXTRACTION

APPROVED

PREPARED BY: P. D. McCart 8/22/66

4.6 Determination of filler when present. If filler has appeared in the extract, the filler weight must be determined as follows:

- 4.6.1 Conditioning of crucibles. Place a glass fiber filter in a fritted glass crucible (see note 6.3). Condition in a forced air oven for 15 to 20 minutes at $325 \pm 5^\circ\text{F}$. Allow the crucibles to cool in a desiccator and weigh to 0.001 gm.
- 4.6.2 Filtering. Set up the vacuum system and filter the resin extract. Using a wash bottle of acetone or appropriate solvent wash out the extraction tube and flask, putting all washings in the filtering crucible. Wash the residue clean of resin. About 50 ml. of acetone or appropriate solvent will usually suffice. Do not stir the solution on the filter.
- 4.6.3 Unremovable filler. If after washing the extraction flask with solvent there is some filler which is caked on the flask and cannot be removed, the following procedure can be used for carbonaceous filled phenolic resins: Place the flask in a muffle furnace at $750 \pm 25^\circ\text{F}$. for a minimum of one hour. Cool in a desiccator and weigh to 0.001 gm. If the flask has been pretared, subtract the tared weight and add this filler weight to that of 4.6.4. If the flask has not been pretared, place in a muffle furnace and burn off the carbonaceous filler at $1000 \pm 50^\circ\text{F}$ for a minimum of one hour. Cool in a desiccator and weigh to 0.001 gm.
- 4.6.4 Drying filtering crucible. Dry the filtering crucible in a forced air oven for a minimum of one hour at $325 \pm 5^\circ\text{F}$. Cool in a desiccator and weigh to 0.001 gm. Add this filler weight to the carrier weight of 4.7.

4.7 Drying thimbles. Remove thimbles from the extraction assembly. Drain off the solvent and dry to essentially constant weight at $325 \pm 5^\circ\text{F}$ in a forced air oven (see note 6.4). If paper thimbles are used, remove from the oven in sets of three or less and weigh immediately to 0.001 gm. (W₄). Glass thimbles may be cooled in a desiccator before weighing.

5. CALCULATIONS AND REPORTING

5.1 Calculations where paper thimbles are used.

5.1.1 Dry resin content (DRC). Calculate dry resin content (resin solids) as follows:

$$\text{DRC, wt. \%} = 100 \left(1 - \frac{W_4 - W_1}{(W_3 - W_2) \left(1 - \frac{V}{100} \right)} \right)$$

where: W₁ = weight of dried thimble, gms.
W₂ = weight of thimble after storage in a desiccator, gms.
W₃ = weight of thimble plus sample, gms.
W₄ = weight of thimble plus extracted sample, gms.
V = volatile content, wt. %

SUBJECT:

D

CHEMICAL TEST METHOD FOR:
RESIN CONTENT BY SOXHLET EXTRACTION

APPROVED

PREPARED BY:

P. D. McCart
P. D. McCart 8/22/66

5.1.2 Wet resin content (WRC). Calculate wet resin content (total pick-up) as follows:

$$\text{WRC, wt. \%} = 100 \left(1 - \frac{W_4 - W_1}{W_3 - W_2} \right)$$

5.1.3 Wet resin solids (WRS). Calculate wet resin solids as follows:

$$\text{WRS, wt. \%} = 100 \left(1 - \frac{W_4 - W_1}{W_3 - W_2} - \frac{V}{100} \right) = \text{WRC} - V$$

5.2 Calculations where glass thimbles are used.

5.2.1 Dry resin content (DRC). Calculate dry resin content (resin solids) as follows:

$$\text{DRC, wt. \%} = 100 \left(1 - \frac{W_4 - W_1}{(W_3 - W_1) \left(1 - \frac{V}{100} \right)} \right)$$

where symbols are as specified in 5.1.1

5.2.2 Wet resin content (WRC). Calculate wet resin content (total pick-up) as follows:

$$\text{WRC, wt. \%} = 100 \left(1 - \frac{W_4 - W_1}{W_3 - W_1} \right)$$




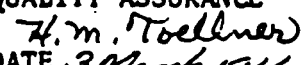
5.2.3 Wet resin solids (WRS). Calculate wet resin solids as follows:

$$\text{WRS, wt. \%} = 100 \left(1 - \frac{W_4 - W_1}{W_3 - W_1} - \frac{V}{100} \right) = \text{WRC} - V$$

5.3 Reporting. Unless otherwise designated, report resin content to the nearest 0.1%. Duplicate determinations should be considered suspect if they differ by more than 1.5%.

6. NOTES

- 6.1 Paper thimbles. When paper thimbles are used, it is good laboratory practice to periodically determine a blank on the procedure. This is especially true when conducting the extraction on a new product for the first time. Under normal conditions, single thimbles shall be used. However, particle size of a filler may make it necessary to use double thimbles.
- 6.2 Selection of solvent. For carbonaceous filled phenolics with carbon or graphite reinforcement, the following rule shall apply: DMF must be used when resin content is less than 31% and/or volatile content is less than 3.8%.
- 6.3 Use of glass fiber filter. A glass fiber filter is used to prevent the fritted glass filter from becoming clogged with the filler.
- 6.4 Thimble drying time. The drying time for the thimble is normally two hours minimum.
- 6.5 Calculation of resin content. This method has used carrier weight for the calculation of resin content. There are occasions where it may be desirable to calculate resin content by using the resin extract which has been dried down by the appropriate means and weighed. Calculating resin content using resin extract typically gives higher results and more variability than when thimble weight is used.

 P.O.S. Chemicals, Inc.		QUALITY ASSURANCE TEST METHODS	DATE 2/11/66	NUMBER CTM-14B Page 1 of 3
SUBJECT: CHEMICAL TEST METHOD FOR: RESIN CONTENT OF PREPREG MATERIALS BY PYROLYSIS <div style="border: 1px solid black; padding: 2px; display: inline-block;">B</div>		APPROVED   PREPARED BY:* P. D. McCart		
APPROVAL SIGNATURES:				
GENERAL MANAGER DATE N/A	ENGINEERING DATE N/A	PRODUCTION DATE N/A	PRODUCTION CONTROL DATE N/A	
PURCHASING DATE N/A	SALES DATE N/A	QUALITY ASSURANCE*  DATE 3 March 1966		

1. SCOPE
 - 1.1 This method describes a procedure for the determination of the dry resin content in molded carbon reinforced phenolic plastics.
2. EQUIPMENT AND MATERIALS
 - 2.1 Equipment.
 - (a) Burners, Fisher
 - (b) Test tubes, vycor, 25 x 200 mm
 - (c) Vacuum source
 - (d) Manometer or vacuum gauge
 - (e) Condenser, cold finger type
 - (f) Clamps
 - (g) Rubber stoppers
 - (h) Vacuum tubing
 - (i) Press for molding carbon bars 1/2" x 1/2" x 5"
 - (j) Desiccator
 - (k) Cutting assembly for cutting carbon bars
 - (l) Balance, analytical
 - (m) timer
3. SAMPLING
 - 3.1 Sample size. A broadgoods sample 6 inches by 20 inches is required.
 - 3.2 Number of determinations. Unless otherwise specified, run duplicate determinations on each sample.
 - 3.3 Preparation of test specimens.
 - 3.3.1 Cutting. Cut 32 rectangles from the prepreg measuring 1/2 inch by 5 inches.
 - 3.3.2 Staging. Stage the 32 rectangles at 255 + 5°F to reduce flow. Staging time is dependent upon resin system and flow and must be determined from experience.
 - 3.3.3 Molding. Using the 32 rectangles, mold a bar 1/2 inch by 1/2 inch by 5 + 1/32 inch. Cure the specimen at 310 + 10°F and 1900 + 100 psi for 15 + 1 minutes. If there is any resin flash, begin again and use a longer staging time. Cut the bar into approximately three equal lengths. Tag and store in a desiccator.

*Signatures necessary for all Quality Assurance Procedures and Instructions.

SUBJECT: CHEMICAL TEST METHOD FOR:
RESIN CONTENT OF PREPREG
MATERIALS BY PYROLYSIS

B

APPROVED

PREPARED BY: P. D. McCart

P. D. McCart

pm

4. PROCEDURE

- 4.1 Setting up apparatus. Set up the apparatus as shown in Figure 1 (see page 3). Weigh the test specimen to 0.01 gms. Place the test specimen in the vycor test tube (see note 6.1) and attach the tube to the side arm of the vacuum flask. All joints must be air tight. Start the vacuum pump, evacuate the system, and check for air leaks by shutting the valve leading to the vacuum pump. The leak rate should be less than five millimeters per minute (0.2 inch) as measured by a manometer or vacuum gauge.
- 4.2 Pyrolysis. With the vacuum on and the pressure stabilized, place the Fisher burners under the specimen so that the flame area covers it completely. Continue pyrolysis for 30 minutes, or until the manometer or vacuum gauge reading is the same as it was before pyrolysis, whichever is longest.
- 4.3 Cooling sample. When pyrolysis is complete, turn off the burners and allow the specimen to cool under vacuum to room temperature. When the specimen is cool, turn the vacuum off, and carefully let air into the system to restore pressure equilibrium.
- 4.4 Reweighing specimen. Remove the vycor test tube from the vacuum flask. Weigh the specimen to 0.01 gm. immediately, or transfer to a desiccator and weigh when convenient.

5. CALCULATIONS AND REPORTING

- 5.1 Calculations. Calculate the dry resin content (DRC) of the sample as follows:

$$\% \text{ DRC} = \left(1.00 - \frac{W_2}{W_1}\right) 100 \times K$$

where: W_1 = weight of sample before pyrolysis, gms.

W_2 = weight of sample after pyrolysis, gms.

K = constant peculiar to material being tested. See Appendix A for appropriate K factor.

- 5.2 Reporting. Unless otherwise specified, round off and report dry resin content to the nearest 0.01% on worksheets and to the nearest unit in the designated place of figures on final reports. Duplicate determinations should be considered suspect if they differ by more than 0.2%.

6. NOTES

- 6.1 Number of specimens per tube. Two or more specimens may be put in a tube so long as all specimens are completely covered by the flame from the burners.

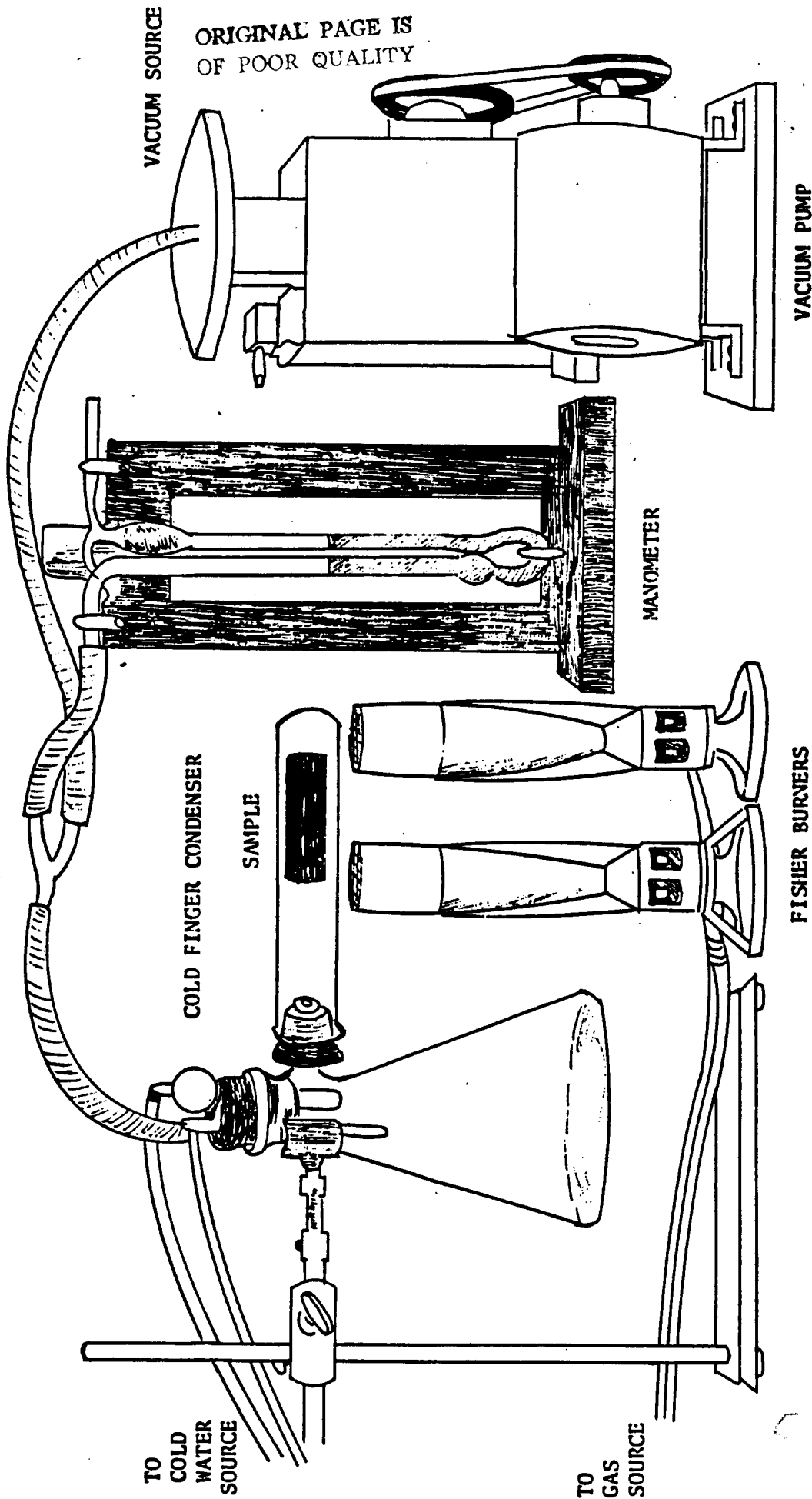
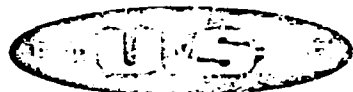


FIGURE 1



Chemicals, Inc.

QUALITY ASSURANCE
TEST METHODS

DATE

11/29/65

NUMBER CTM-18A

Page 1 of 3

SUBJECT:

CHEMICAL TEST METHOD FOR:
DEGREE OF POLYMERIZATION
BY ACETONE EXTRACTION

APPROVED

PREPARED BY: * P. D. McCart

P. D. McCart

A

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING
DATE N/ASALES
DATE N/AQUALITY ASSURANCE*
H. M. Toellner
DATE 12-3-65

1. SCOPE

- 1.1 Scope. This method describes a procedure for determining the amount of acetone-soluble material in prepreg, molded or laminated phenolic products.
- 1.2 Equivalent methods. This method is equivalent to Federal Test Method Standard No. 406, Method 7021 and ASTM-D494-46.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Specimen fragmentator - a suitable drill press, bench lathe, planer, milling machine, grinder, or file will be needed to break up specimen into fragments
- (b) Sieves, U. S. Standard, 40 and 140 mesh
- (c) Weighing bottle, 2 oz. glass stoppered
- (d) Soxhlet extraction assembly, 125 ml. capacity with hot plate
- (e) Balance, analytical
- (f) Drying chamber capable of maintaining $122 \pm 4^{\circ}\text{F}$

2.2 Materials.

- (a) Extraction thimble, paper, 25 mm x 80 mm
- (b) Aluminum dishes, diameter 2-1/2 inches, height 1-1/16 inches
- (c) Acetone, reagent grade

3. SAMPLING

- 3.1 Sample size. Each sample shall weigh a minimum of 15 grams. The sample shall be a composite, representative of the prepreg, molded or laminated part from which it was taken.
- 3.2 Number of determinations. Unless otherwise specified, make three determinations on each sample.

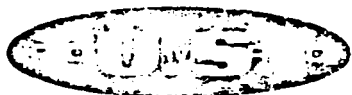
4. PROCEDURE

4.1 Preparation of test specimen.

- 4.1.1 Prepreg. Prepreg materials shall be cut in squares no larger than 1/2 inch.
- 4.1.2 Molded or laminated parts. Molded or laminated parts shall be prepared as follows:

ORIGINAL PAGE IS
OF POOR QUALITY

*Signatures necessary for all Quality Assurance Procedures and Instructions.



Chemicals, Inc.

QUALITY ASSURANCE
TEST METHODS

DATE

11/29/65

NUMBER CTM-18A

Page 2 of 3

SUBJECT:

CHEMICAL TEST METHOD FOR:
DEGREE OF POLYMERIZATION
BY ACETONE EXTRACTION

A

APPROVED

P. D. McCart

PREPARED BY: P. D. McCart

4.1.2.1 Grinding. The method of grinding up the material for sieving should be such that no undue heating of the particles will occur which might tend to further cure the material. Drilling is the preferred method where the size and shape of the part permits. In any case, the particles should be of the smallest size practicable, so that they will pass through a 40-mesh screen with a minimum of reworking or grinding.

4.1.3 Screening. Screen the material through a 40-mesh sieve. Regrind the part which does not pass through the 40-mesh sieve until it does, resieve, and blend with the original part which passes. Place the sample on a 140-mesh sieve and screen by using either a standard screen shaker (such as the Rotap) or by hand screening. In the latter case, the screen should be rotated with a slight tapping for a period of one minute or more until there are virtually no further screenings.

4.1.4 Bottling sample. The sample for analysis should be removed from the 140-mesh screen and placed in the two-ounce bottle. Care should be taken that the sample is immediately placed in this bottle and closed up so as to prevent absorption of moisture by undue exposure.

4.2 Extraction. From the material in the bottle, accurately weigh out 1.0 ± 0.5 gm. into a tared, open texture, quantitative filter paper 12 to 15 cm. in diameter. Place the filter paper containing the test specimen in a standard, single thickness, paper extraction thimble, 80 mm by 25 mm. Close the thimble so that none of the sample can float out. Place the thimble in an extraction tube and add 50 ml. of acetone. Start the water in the condenser and adjust the heat so there are between 15 and 20 dumps per hour. Carefully maintain this rate for four hours minimum. After the siphon empties, remove the flask and empty the contents into a weighed aluminum dish. Rinse the flask with acetone three times and add the washings to the extract in the dish.

4.3 Drying. Place the dish in a well ventilated drying chamber maintained at $122 \pm 4^\circ\text{F}$ and dry the sample to constant weight. Keep the dish in a desiccator between dryings to prevent moisture pickup.

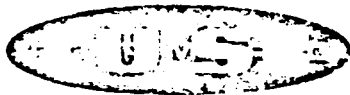
5. CALCULATIONS AND REPORTING

5.1 Calculations.

5.1.1 Acetone extractables. Calculate the percent of acetone extractable material as follows:

$$E = \frac{W_3 - W_2}{W_1} \times 100$$

where: E = acetone extractables, wt. %
W₁ = sample weight, gms.
W₂ = tare weight of aluminum pan, gms.
W₃ = final weight of pan and extract, gms.



Chemicals, Inc.

QUALITY ASSURANCE
TEST METHODS

DATE

11/29/65

NUMBER CTM-18A

Page 3 of 3

SUBJECT:

CHEMICAL TEST METHOD FOR:
DEGREE OF POLYMERIZATION
BY ACETONE EXTRACTION

APPROVED

PREPARED BY: P. D. McCart

P. D. McCart

5.1.2 Percent uncured resin. Calculate percent uncured resin as follows:

$$U = \frac{E}{T}$$

where: U = uncured resin, wt. %

T = resin solids, wt. % (see note 6.1)

5.1.3 Degree of polymerization. Calculate degree of polymerization as follows:

$$\text{Degree of polymerization} = 100 - U$$

5.2 Reporting. Unless otherwise specified, report the following information:

- (a) Percent acetone extractable material
- (b) Degree of polymerization
- (c) Cure cycle used in preparation of part

6. NOTES

6.1 The resin solids figure must be representative of the resin solids of the sample extracted.



QUALITY ASSURANCE
TEST METHODS

DATE
12/16/82

NUMBER
CTM-21C
Page 1 of 4

SUBJECT:
CHEMICAL TEST METHOD FOR:
DEGREE OF ADVANCEMENT OF PHENOLIC
RESINS BY INFRARED SPECTROPHOTOMETRY

PREPARED BY: *James B. Rock*
James B. Rock

1.0 SCOPE

1.1 This method describes a procedure for the determination of the degree of advancement or polymerization of phenolic resins. An infrared analytical technique is employed which differentiates molecular structural changes resulting from polymerization. This method applies to raw resin as well as preimpregnated fabric, to unmodified as well as modified resins, and to filled as well as unfilled preregs.

2.0 EQUIPMENT AND AMATERIALS

2.1 Equipment

- (a) Infrared spectrophotometer, Beckman IR-8, or equivalent
- (b) Sodium chloride variable path cell
- (c) Sodium chloride liquid cell, 0.20 mm
- (d) Funnel, 60°
- (e) Beakers, 100 ml
- (f) Stirring rod, glass
- (g) Scissors
- (h) Graduated cylinder, 50 ml
- (i) Syringe, 2 ml
- (j) Centrifuge
- (k) Vacuum oven
- (l) Aluminum pans, 2 1/2" diam. 1/2" height

2.2 Materials

- (a) Acetone, water free, saturated with silica gel
- (b) Filter paper, S & S No. 589 Blue Ribbon, or equivalent
- (c) Infrared chart paper, 2.5 to 16 microns, absorbance

3.0 SAMPLING

3.1 Sufficient sample shall be taken to provide approximately 2 grams of resin solids.

4.0 PROCEDURE

4.1 Operation and Calibration

For operation and calibration of the spectrophotometer, see SCM-7-01 for the IR-8 instrument or other equivalent procedures.

4.2 Adjustment of variable cell

The variable path cell shall be adjusted to the exact thickness of the 0.20 mm sodium chloride cell. In order to determine that the thicknesses are closely matched,



QUALITY ASSURANCE
TEST METHODS

DATE
12/16/82

NUMBER
CTM-21C
Page 2 of 4

SUBJECT: CHEMICAL TEST METHOD FOR:
DEGREE OF ADVANCEMENT OF PHENOLIC
RESINS BY INFRARED SPECTROPHOTOMETRY

PREPARED BY: •

James B. Rock

4.2 fill both cells with acetone using a syringe. When properly matched, the resulting spectrum will not vary by more than $\pm 3.0\%$ from 100.0% transmission when measured between 9.5 and 15.0 microns. Adjust the variable path cell as necessary in order to attain the above tolerance.

4.3 Solution Preparation

4.3.1 Raw Resins Raw resins known or suspected of having solvents other than acetone shall be devolatilized under vacuum without heat for a minimum of four hours or until disappearance of the absorption bands indicating their presence. This may require as long as 24 hours for some solvents such as isopropanol. (As an alternate, isopropanol can be devolatilized by adding 30 ml acetone to 2 gm phenolic resin and periodically stirring at RT without vacuum over 0.5 hr during which both acetone and isopropanol are largely devolatilized). Add sufficient acetone to the resin to effect a 4 to 6% solids solution. Stir with a glass stirring rod and allow to stand for ten minutes. If the solution is not clear, centrifuge, or filter using S & S No. 589. Seal filtrate in a vial if there is to be any time delay. Continue per instruction in Para. 4.4.

4.3.2 Preimpregnated Resins Cut the sample into approximately 1/2 inch squares or lengths. If the dry resin content of the prepreg is known, use the following formula to determine amount of prepreg and acetone to use:

$$\frac{191}{\text{DRC}} = \text{grams of prepreg in 36 ml of acetone}$$

If the dry resin content of the prepreg is not known, arbitrarily use 6 grams of prepreg in 36 ml of acetone. Place the prepreg and acetone in a 100 ml beaker. Stir with a glass rod and allow to stand for a minimum of 20 minutes with occasional stirring. Filter using S & S No. 589. Seal filtrate in a vial if there is to be any time delay. Extraction efficiency should be determined. Extraction efficiency must be at least 90% (see Para. 6.1) in order to achieve an accurate result.

4.4 Adjustment of Concentration Fill the 0.20 mm cell with the resin-extract from Para. 4.2, using a syringe. Fill the variable path cell with water free acetone. Position both cells in their respective holders in the spectrophotometer. Without placing the pen upon the chart paper,



QUALITY ASSURANCE
TEST METHODS

DATE 12/16/82

NUMBER CTM-21C
Page 3 of 4

SUBJECT: CHEMICAL TEST METHOD FOR:
DEGREE OF ADVANCEMENT OF PHENOLIC
RESINS BY INFRARED SPECTROPHOTOMETRY

PREPARED BY: *

James B. Rock

- 4.4 start machine operation at 11 microns by setting to "Scan" position. Determine absorbance at approximately 12.15 microns (12.1 to 12.2 microns). The concentration of the extract must be further adjusted (if necessary) to produce linear absorbance between 40% and 45% at this wavelength. This may be accomplished by removing the sample and adding acetone (if the absorbance is too great) or by partially devolatilizing the acetone at room temperature (if the absorbance is too small).
- 4.5 Scan Place the pen to 5.0 microns and place on chart paper. Start machine operation by setting to scan position. Scan the spectrum from 5.0 to 16.0 microns at 0.54 microns per minute. The pen will automatically raise off the chart when the scan is complete. Remove the chart and label it in accordance with Para. 5.2. Turn function control to "Stop" and return pen carriage to 2.5 microns. Tilt pen backwards to prevent accidental contact with metallic chart bed.
- 4.6 Clean Up Remove cells from the instrument. Clean thoroughly with acetone followed by dry nitrogen or dry air. Place the cleaned and dried cells in a desiccator.
- 5.0 CALCULATIONS AND REPORTING
- 5.1 Calculations Calculate the Advancement Index as follows:
- 5.1.1 Construction of tangential base line Construct tangential base line over the absorbance occurring at approximately 9.8 microns, extending from approximately 9.45 to 10.2 microns. Also construct a tangential base line over the absorbance occurring at approximately 12.15 microns extending from approximately 11.5 to 12.5 microns.
- 5.1.2 Construction of perpendiculars Construct a line perpendicular to the wavelength at the absorptions occurring at approximately 9.8 to 12.15 microns which passes through the maximum absorbance and the tangential base line.
- 5.1.3 Determination of absorbance Determine the absorbance units (to three significant figures) between the maximum absorbance and the tangential base line at the approximate 9.8 and 12.15 microns absorptions. This is the base line absorbance.
- 5.1.4 Advancement Index The Advancement Index, Infrared Polymerization Index (IRPI) and IRZB are as follows:



QUALITY ASSURANCE
TEST METHODS

DATE 12/16/82

NUMBER CTM-21C

Page 4 of 4

SUBJECT: CHEMICAL TEST METHOD FOR:
DEGREE OF ADVANCEMENT OF PHENOLIC
RESINS BY INFRARED SPECTROPHOTOMETRY

PREPARED BY:*

James B. Rock

$$\text{Advancement Index (IRPI)} = \frac{\text{Base Line Absorbance @ 12.15 Micr.}}{\text{Base Line Absorbance @ 9.8 Microns}}$$

$$\text{Base Line (IRZB)} = \frac{\text{Log Base Line @ 12.15 Microns}}{\text{Log Base Line @ 9.8 Microns}}$$

5.2 Reporting Record and report the following information:

- (a) Spectrum number
- (b) Sample identification
- (c) Lot or batch number
- (d) Date of test
- (e) Gain, balance and speed settings
- (f) Advancement Index (IRPI) or Baseline Index (IRZB) to the nearest 0.01 unit
- (g) Identity of the operator
- (h) Extraction efficiency if calculated

6.0 NOTES

- 6.1 Extraction Efficiency It is desirable to determine extraction efficiency. This can be done by a variety of normal laboratory techniques.

SUBJECT: CHEMICAL TEST METHOD FOR:
pH OF RAW FABRICS

APPROVED

PREPARED BY: * P. D. McCart

P. D. McCart

APPROVAL SIGNATURES:

GENERAL MANAGER N/A DATE	ENGINEERING N/A DATE	PRODUCTION N/A DATE	PRODUCTION CONTROL N/A DATE
PURCHASING N/A DATE	SALES N/A DATE		QUALITY ASSURANCE* <i>H. M. Tollner</i> DATE 9 DEC. 1965

1. SCOPE

- 1.1 This procedure describes two methods for the determination of pH of raw fabrics. Method I describes a procedure for the determination of pH using cold water extraction. Method II describes a procedure for the determination of pH using hot water extraction.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Pan balance, minimum sensitivity 0.1 gm.
- (b) Beakers, 150 ml. tall form
- (c) pH meter, Corning model 7 or equivalent
- (d) Watch glasses, 65 mm. (2-1/2")
- (e) Hot plate

2.2 Materials.

- (a) Distilled water, freshly boiled, pH 7.00 ± 0.05

3. SAMPLING

- 3.1 Sample size. The test requires a representative sample of at least 8 gms.
3.2 Number of determinations. Unless otherwise specified, conduct duplicate determinations on each sample.

4. PROCEDURE

4.1 Method I. Cold water extraction of fabrics for determination of pH.

- 4.1.1 Extraction. Weigh 4.0 ± 0.2 gms. of fabric, cut to approximately 1/2" x 1/2" pieces, into a 150 ml. beaker. Add 100 ± 2 ml. of room temperature freshly boiled distilled water with a pH of 7.0 ± 0.05 . Stir the fabric-water mixture and ascertain that the fabric has been thoroughly wetted. Cover with a watch glass and allow to stand at room temperature for 60 ± 5 minutes.
- 4.1.2 Determination of pH. If necessary, calibrate the pH meter in accordance with SCM-7-02. Rinse the electrodes with distilled water and immerse the electrodes about one inch in the solution to be tested. Turn the instrument to the "read" position and allow 15 seconds to pass before taking a reading. Record the reading to the nearest 0.02 pH unit.

*Signatures necessary for all Quality Assurance Procedures and Instructions.

SUBJECT:

CHEMICAL TEST METHOD FOR:
pH OF RAW FABRICS

APPROVED

P. D. McCart

Im

PREPARED BY: P. D. McCart

(B)

4.2 Method II. Hot water extraction of fabrics for the determination of pH.

4.2.1 Extraction. Weigh 4.0 ± 0.2 grams of fabric, cut to approximately $1\frac{1}{2}'' \times 1\frac{1}{2}''$ pieces, and transfer to a 200 ml. beaker. Add 100 ± 2 ml. of freshly boiled, distilled water with a pH of 7.0 ± 0.05 . Cover with a watch glass and boil on a hot plate for 20 to 30 minutes. Remove from the hot plate and allow the solution to reach room temperature. Add freshly boiled, distilled water with a pH of 7.0 ± 0.05 to bring the volume to 100 ± 2 ml. and determine the pH as in 4.1.2.

5. CALCULATIONS AND REPORTING

5.1 Report the average of two tests to 0.01 pH unit. Duplicate determinations should be considered suspect if the range exceeds 0.5 pH unit.

pH VALUE - BOILING WATER

NASA 71108/71109 (filler, fabric) per CTM 246 and ASTM D1512-84

Equipment

- Fisher Electrometer Model 380

Sampling

- Filler - as given
- Fabric - cut in 1-1/2" x 1-1/2 squares
- 5 mg of sample per beaker

Procedure

- Begin by standardizing pH meter with 4.0, 7.0, 10.0 standard pH solutions
- Weigh 5 mg of sample into a 100 ml beaker
- Add a few drops of methanol which acts as a disperant
- Add 50 mls of DI H O (which has a pH of 7.0
- Cover with watchglass or foil to enhance boiling and prevent evaporation
- Bring to slow boil for thirty minutes
- Cool to room temperature
- Do duplicate determinations on each prepared sample and average
- Standardize at time of testing
- If consistant numbers are recorded - Check pH standards, check pH of water used, resample and retest



QUALITY ASSURANCE
TEST METHODS

DATE

3/4/82

NUMBER CTM-49A

Page 1 of 5

SUBJECT: CHEMICAL TEST METHOD FOR:
LIQUID CHROMATOGRAPHY & GEL PERMEATION
OF PHENOLIC RESINS

PREPARED BY: *

James Rock & F. Bancroft

1.0 SCOPE

1.1 This procedure describes methods for conducting liquid Chromatography and gel permeation chromatography of phenolic resins. It analyzes the moleweight constituents.

2.0 EQUIPMENT AND MATERIALS

- 2.1 HPLC Spectra-Physics SP8000 with UV detector and autosampler.
- 2.2 μ -Styragel chromatography columns, 2 x 100 A, 2 x 500 A
(USE NO WATER)
- 2.3 Syringe, 10cc
- 2.4 Gas tight syringe, 5cc
- 2.5 Needles
- 2.6 Scissors
- 2.7 Centrifuge tubes, 50 ml disposable
- 2.8 Centrifuge
- 2.9 Graduated cylinder, 10 m.
- 2.10 Balance
- 2.11 Beaker
- 2.12 Millex-SR filter units, 0.5 μ m
- 2.13 Membrane TFE filter paper, 0.45 μ m
- 2.14 B&J tetrahydrofuran, UV grade
- 2.15 Helium (zero grade) gas
- 2.16 Nitrogen gas

3.0 PREPARATION OF SAMPLE SOLUTION

- 3.1 Resin Samples
 - a. Weigh out 0.25 \pm 0.02 gm of resin into a glass vial (labeled)
 - b. Add 10 ml 100% THF into the vial
- 3.2 Resin Mixes
 - a. Weigh out 3.0 \pm 0.02 gm of sample into a centrifuge tube
 - b. Add 25.0 ml of 100% THF into the centrifuge tube
 - c. Agitate for 15-50 minutes
 - d. Centrifuge for 2-5 minutes (use speed-7)
- 3.3 Prepregs
 - a. Cut sample into approximate 1/2" x 1/2" squares
 - b. Weigh out 6.0 \pm 0.5 gm of sample into centrifuge tube
 - c. Add 25 ml 100% THF into the centrifuge tube
 - d. Agitate for 15-50 minutes
 - e. Centrifuge for 2-5 minutes (Speed-7)
- 3.4 Solvent Preparation
 - a. Filter solvent (1-2 liters) by using 0.45 μ m TFE filter paper
 - b. Degas with He 1/4 to 1/2 hour (high flow)
 - c. Decrease the flow rate before running

4.0 INSTRUMENT PROGRAMING

4.1 Date Assignment

D: 00:00:00 00:00:00
 month day year hour min. sec.

D - Display



QUALITY ASSURANCE
TEST METHODS

DATE

3/4/82

NUMBER

CTM-49A

Page 2 of 5

SUBJECT:

CHEMICAL TEST METHOD FOR:
LIQUID CHROMATOGRAPHY & GEL PERMEATION
OF PHENOLIC RESINS

PREPARED BY:*

James Rock & F. Bancroft

4.2 Report File Assignment

R:1 - report file

REPORT FILE 1

RENAME:

SAMPLE NAME:-----

SAMPLE VOLUME: 10 μ L

SAMPLE CONC.: 20 MG-ML

OPERATOR NAME: (your name)

DETECTOR 1: UV. 254NM 0.1 AUFS

DETECTOR 2: ;

COLUMN TYPE; SIZE: 2x100A, 2X500A, μ -STYRAGEL

MOBILE PHASE: Isocratic

A: ;

B: ;

C: THF

END OF DIALOG

4.3 Parameter File Assignment:

S:1 parameter file 1

PARAMETER SET: 1

RENAME: ;

1. LINK: ;
2. INITIAL DELAY: 1
3. RUN TIME: 30
4. NO. OF CYCLES: 6
5. FLOW RATE: 2.00
6. FLOW MODE: QI
7. TEMPERATURE: 35
8. MOBILE PHASE FILE: 1
9. REPORT FILE: 1
10. GRAPH FILE: 1
11. DATA SYSTEM FILES: 1
12. CHAN 1:1
13. CHAN 2: ;
14. FILL TIME: 1
15. FLUSH TIME: 1
16. REPETITIONS PER VIAL: 1
17. TIMED EVENTS: XX

END OF DIALOG

4.4 Graph File Assignment

G = 1 graph file 1

G11
G1E5
G1I5
GVO.5
G1L
XX

EXIT GRAPH EDIT



QUALITY ASSURANCE
TEST METHODS

DATE

3/4/82

NUMBER

CTM-49A

Page 3 of 5

SUBJECT:

CHEMICAL TEST METHOD FOR:
LIQUID CHROMATOGRAPHY & GEL PERMEATION
OF PHENOLIC RESINS

PREPARED BY: •

James Rock & F. Bancroft

4.5 Mobile Phase File Assignment

M = 2 mobile phase file 2

MOBILE PHASE FILE 2

WHICH SOLVENTS? C

END OF DIALOG

MI2

M;l

MOBILE PHASE FILE 1

WHICH SOLVENTS? C

END OF DIALOG

MI1

4.6 Data File Manipulation:

FI1 Data system file (file center)

nm gpc-QC

CH 001

OD 002

IX 001

RN 001

NR 001

PW 015

PT 0150

MA 5000

FD

VE

FILE 001

RN 001

NR 001

PW 015

PT 0105

FS 000

MS 0

MA 5000

IM 00

PP 004

T1 000

RO 000

NM GPC-QC

CH 001

OD 002

IX 001

SN 000

FF function file

APO

FD

VE

FILE 001

QUALITY ASSURANCE
TEST METHODS

DATE

3/4/82

NUMBER

CTM-49A

Page 4 of 5

SUBJECT:

CHEMICAL TEST METHOD FOR:
LIQUID CHROMATOGRAPHY & GEL PERMEATION
OF PHENOLIC RESINS

PREPARED BY: *

James Rock & F. Bancroft

AR 0	IR 0	DC 0	NZ 0	FX 0	EC 0
TP 1	NP 0	NT 0	AA 0	XD 0	HD 0

XX

METHOD o

4.7 Review File Values

S-1

PARAMETER SET: 1

INITIAL DELAY: 1 RUN TIME: 30 NO. OF CYCLES: 6

FLOW RATE: 2.00 FLOW MODE: Q1 TEMPERATURE: 35

MOBILE PHASE FILE: 1 REPORT FILE: 1 GRAPH FILE: 1

CHAN 1: 1

FILL TIME: 1 FLUSH TIME: 1 REPETITIONS PER VIAL: 1

R-1

-----VOL: 10 UL CONC: 20 mg-ML NO: 1

(your name)

FLOW RATE: 2.0 PRESSURE: 0000

COLUMN: 2x100A, 2X500A, μ -STYRAGELCHAN 1: U.V. 254NM 0.1 μ FS

MOBILE PHASE: ISOCRATIC

MOBILE PHASE FILE 1

100.0% C

G-1

Graph File: 1

Channel 1: Plot Var.: DET. 1 Chart Spd.: 0.5

Polar: L ATTN.: 5 Zero: 0% Inten.: 5

CHANNEL 2: Inoperative

M-1

Mobile Phase File: 1

100.0% C

5.0 PROCEDURE

5.1 HPLC: Start-up:

- 5.1.1 Turn on He and N₂
- 5.1.2 Adjust the He pressure to 10 psi and N₂ pressure to 75 psi
- 5.1.3 Turn on both switches

QUALITY ASSURANCE
TEST METHODS

DATE

3/4/82

NUMBER

CTM-49A

Page 5 of 5

SUBJECT:

CHEMICAL TEST METHOD FOR:
LIQUID CHROMATOGRAPHY & GEL PERMEATION
OF PHENOLIC RESINS

PREPARED BY:•

James Rock & F. Bancroft

Wavelength Drive at "on" position

Set wavelength at 254 nm

- 5.1.4 Turn on the Spectrophotometric Detector, make sure it is ".4 m amp" Set range at 0.1 AUF

5.2 Automatic Operation

- 5.2.1 Be sure both detector and auto sampler are on

- 5.2.2 Keep He purge, the solvent with low flow rate

- 5.2.3 Type AT1
DID
FM1
MIL
SBI

- 5.2.4 Use the inner knob on the detector range selector to adjust the read out to - 1200 (detector signal) or type GB and adjust the baseline 1 inch to the left margin, then type GX to stop plotting..

- 5.2.5 Fill one gas tight syringe with sample solution and the other one with THF; attach each syringe to one Miller-SR filter unit.

- 5.2.6 Place the injector handle in the "LOAD" position and type SO to open the injector sample solenoid

- 5.2.7 Inject 1 cc of THF to flush the injector loop, then inject 1 cc of sample solution and type SK to close the sample solenoid

- 5.2.8 Wash the syringe with the THF twice; fill it with the next sample.

- 5.2.9 After LC, inject automatically, replace the injector handle in the "LOAD" position and repeat the injecting operation.

- 5.2.10 Check the condition by typing:

E - elapsed time since injection
T - present oven temperature
P - pressure
C - current % C at ternary valve
DID - detector signal
F - flow rate

5.3 Calibration (before running sample solutions)

- 5.3.1 Run a THF blank in order to plot the baseline

- 5.3.2 Run molecular weight standards and plot molecular weight vs. retention time on graph paper

Mole Weight Standards:

M.W. Compound
93 Toluene
570 Polystyrene
955 "
3600 "

5.4 Termination

- 5.4.1 Type EX to end run

- 5.4.2 Type SX to stop parameter set

- 5.4.3 Shut down all gas flows

NASA 71108/71109 RESIN

CTM-49A

Equipment

- Beckman 112 Pump
- 160 Detector
- Ultrastyagel 500 A Column
- 254 nm/0.1 Au Detector

Procedure

- Solvent prefiltered with Alpha 200 0.2 um membrane filter followed by degassing for thirty minutes with Helium
- Measured 50 mg sample and added 20 ml of THF (C = 5.0 mg/ml)
- Agitate for ten minutes
- Samples containing carbon black were centrifuged for five minutes
- To prevent any large particles from entering through the system an Quodise 0.2 micron filter was attached to the syringe
- The samples were then injected using a 10 ul sample loop
- GPC method on the Nelson Programs and GPC PHEN. for calibration was used to obtain the GPC report (run time: 10 min)

NOTE Calibration report attached.

ORIGINAL PAGE IS
OF POOR QUALITY

*** Calibration Data ***

Calibration Name:

Misc Information:

Fit Type: 3

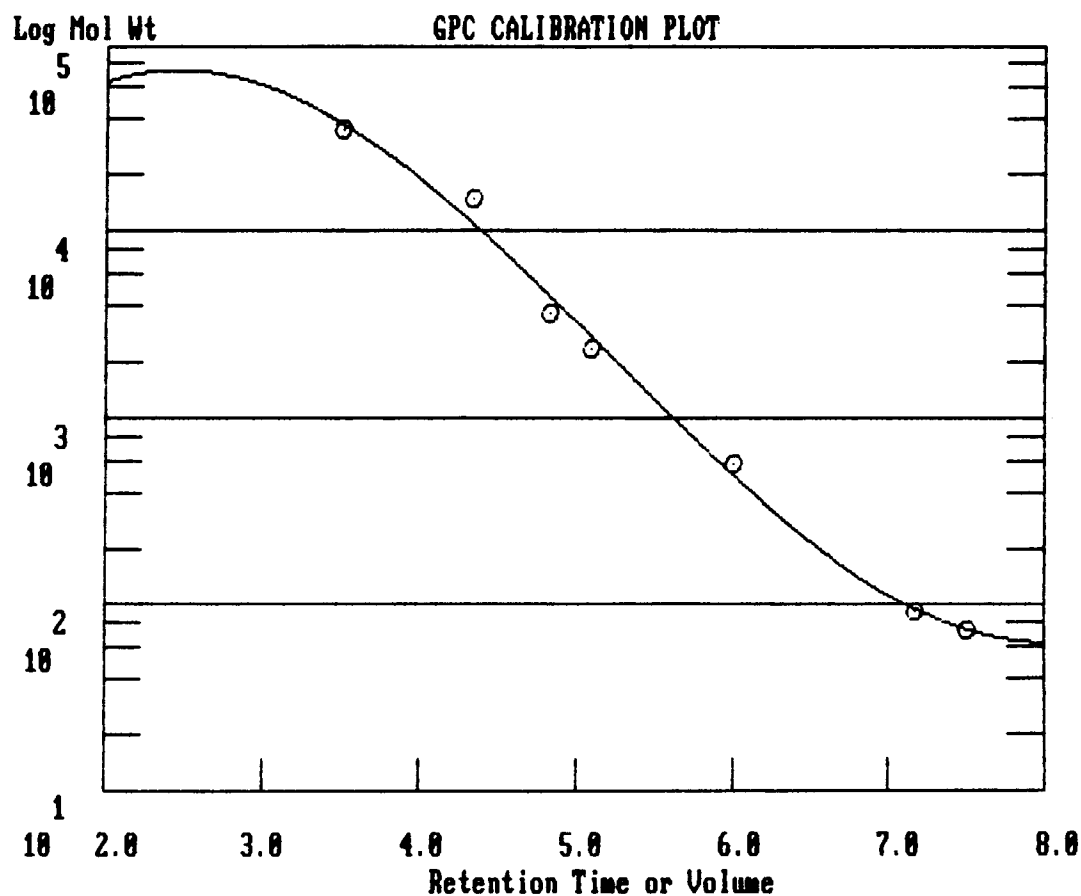
Log Mol Wt = $A + Bx + Cx^2 + Dx^3$

A= 2.538977 B= 2.115815 C= -.5646824 D= 3.606432E-02

Coefficient of Determination: 0.9902

Ret Time	Molecular Weight	Log Mol Wt
----------	------------------	------------

3.50	35000	4.544
4.33	15000	4.176
4.83	3600	3.556
5.09	2350	3.371
6.00	570	2.756
7.17	92	1.964
7.50	72	1.857



QUALITY ASSURANCE
TEST METHODSDATE
4/1/82NUMBER
CTM-50A
Page 1 of 2

SUBJECT:

CHEMICAL TEST METHOD FOR:
DIFFERENTIAL SCANNING CALORIMETRY

PREPARED BY:

James B. Rock

1.0 SCOPE

1.1 This procedure describes a method for conducting differential scanning calorimetry.

2.0 EQUIPMENT AND MATERIALS

- 2.1 Perkin-Elmer DSC-1B or equivalent analyzer
- 2.2 Sample pans, crimped
- 2.3 Nitrogen gas supply

3.0 SAMPLING

3.1 Obtain a small (1 oz) representative sample.

4.0 PROCEDURE

4.1 Perform test with a Perkin-Elmer DSC-1B or equivalent analyzer. Sample pan must be crimped.

Temperature range: Room temp. to 325°C (598°K)

Rate of heating: 20°C/minute

Sensitivity range: 10 mcal/sec. (20 mvfs)

Reference: Empty gold pan plus lid on reference side

Atmosphere: Nitrogen (40 ml/min)

4.2 The peak area is determined by extrapolating the baseline under the peak. Measure the peak with a planimeter or equivalent. Record the temperature of onset, peak (s), maximum, and completion of reaction. The heat of polymerization shall be calculated as follows:

$$H_s = \frac{H_r \times W_r \times A_s \times R_s \times S_r}{W_s \times A_r \times R_r \times S_r}$$

where:

H = heat of polymerization, cal/gm

W = weight, mg

A = peak area, in²

S = chart speed, in/sec.

R = range setting, mcal/sec - in

Subscripts:

r = reference (calibration standard)

s - sample



QUALITY ASSURANCE
TEST METHODS

DATE

4/1/82

NUMBER

CTM-50A
Page 2 of 2

SUBJECT:

CHEMICAL TEST METHOD FOR:
DIFFERENTIAL SCANNING CALORIMETRY

PREPARED BY: *

James B. Rock

4.3 Melting Point Determinations

1. Polymeric materials and organic solids melt points may be determined by the apex of the peak of melt endotherm.
2. Pure metal melt points may be determined as the onset of the melt endotherm.

DSC

NASA 71108/71109 (RESIN AND PREPREG) PER CTM 50-A

Equipment

- Perkin Elmer DSC-2
- DuPont 990

Sampling

- Resin - 3 - 5 mg (devolatilized) in crim aluminum pan
- Prepreg - 10 - 15 mg in open aluminum pan

Procedure

- Temperature Range - R.T. to 325 C
- Heating Rate - 10 C/min
- Sensitivity - 5.0 m cal/sec
- Atmosphere - Nitrogen - 40 ml/min
- Place sample pan on rear
- Place sample pan on rear platform (for DuPont)
- Place sample pan on left cell (for P&E)
- Place reference pan on front platform (for DuPont)
- Place reference pan on right cell (for P&E)
- Begin heating and recording
- Cool chamber with air only
- Draw tangents to determine peak exotherm
- Use recent calibration report to calculate final temperature with ΔT .
- Calibration was performed weekly
- If pen becomes noisy - check nitrogen tank for good pressure
- Check flow rate
- Check to make sure pans are properly placed



QUALITY ASSURANCE
TEST METHODS

DATE

3/6/80

NUMBER

CTM-51

Page 1 of 2

SUBJECT:

CHEMICAL TEST METHOD FOR:
Thermogravimetric Analysis

PREPARED BY:

James B. Rock
James B. Rock

1.0 SCOPE

1.1 This procedure describes the method for conducting thermogravimetric analysis of resins and prepreg.

2.0 EQUIPMENT AND MATERIALS

- 2.1 Perkin-Elmer Model TGS-2 Thermoanalyzer or equivalent
- 2.2 Perkin-Elmer DSC-2 Programmer, or equivalent

3.0 SAMPLING

3.1 Obtain a small (1 oz.) representative sample

4.0 PROCEDURE

4.1 Balance Controls

- 4.1.1 Power - on
- 4.1.2 Filter - lo
- 4.1.3 Recorder Mode - short (see sec. 7D1,6 and 7D2,1)
- 4.1.4 Recorder Range - 10 mg
- 4.1.5 Weight Suppression switch - 100 mg
- 4.1.6 Weight Suppression thumb wheel dial - 00.000

4.2 Use DCS-2 as the TG Programmer:

- 4.2.1 Sample holders empty
- 4.2.2 Temperature RANGE Calibration - as calibrated
- 4.2.3 Temperature ZERO Calibration - as calibrated
- 4.2.4 Lower LIMIT - 350°K (or as required)
- 4.2.5 Cooling RATE - 160°/min.

4.3 Heater Control Unit

- 4.3.1 ZERO and RANGE Setting - as determined from calibration procedure (or as indicated on calibration card 993-9235)
- 4.3.2 Power - HEAT
- 4.3.3 °C - °K switch (on back panel) °K

4.4 Recorder

- 4.4.1 CHART SPEED - slow (e.g., 5 mm/min.) change to faster chart speed when temperature program is started
- 4.4.2 Weight Pen: RANGE - 20 mv; right-hand zero
- 4.4.3 Second Pen: Thermocouple: RANGE - 1 volt, right-hand zero; or FDC: RANGE - 10 mv, left-hand zero



QUALITY ASSURANCE
TEST METHODS

DATE
3/6/80

NUMBER CTM-51
Page 2 of 2

SUBJECT:
CHEMICAL TEST METHOD FOR:
Thermogravimetric Analysis

PREPARED BY: •
James B. Rock

4.5 FDC

4.5.1 RANGE - "S" (standby) change to range 2 after all other settings have been made and the sample has equilibrated

4.5.2 FILTER - Medium

4.5.3 FDC Pen: RANGE 20 mv



QUALITY ASSURANCE
TEST METHODS

DATE 5/7/84

NUMBER CTM-51
Appendix A
Page 1 of 2

SUBJECT:

CHEMICAL TEST METHOD FOR:
THERMOGRAVIMETRIC ANALYSIS

PREPARED BY: •

D. San Miguel

1.0 MATERIAL: CCA-3 Fabric (512°C Test)

2.0: EQUIPMENT: Setting

2.1 Heater Control: Load
Zero - + 20
Range - + 10
TGS - 2

2.2 Auto Balance:
Power - On
Filter - LO
Tare - Pointer at the middle of green line
Recorder Range - 100
Recorder Mode - Short

2.3 Recorder:
Pen 2 - (Zero pen by holding; zero switch down; adjusting knob until pen is "0" on the right hand side of the chart)
Range - 1 volt
Power - Servo
Pen 1 (Zero by following instruction of Pen 2)
Range - 1 mv
Polarity - (-)
Chart Speed - 5 mm/min.

2.4 Differential Scanning Calorimeter
Power - On
Lower - Unit - 100°C
Upper Limit - 512 ± 10°C
Cooling Range - 160°C/min.
Heating Rate - 160°C/min.

2.5 Air flow rate - 80 ml/min (Zero gas air)

3.0 TEST METHOD

- 3.1 Cut three 1/4" x 1/4" of fabric
- 3.2 Disengage spring latch of balance cover to release gas pressure
- 3.3 Loosen screw, then lower down furnace
- 3.4 Swing loading platform underneath platinum pan
- 3.5 Place samples on pan
- 3.6 Raise furnace carefully, tighten screw, and engage spring latch (Note; bubbles should appear on the outlet-tubing that is immersed in water; if not, turn balance cover until bubbles come out)
- 3.7 Turn heater control to heat and recorder mode to Mg
- 3.8 Make mark on chart by lowering Pen-1 and advancing chart manually and raising pen again (reading will be 1 mg per small division, this will give the weight of sample).



QUALITY ASSURANCE
TEST METHOD

DATE 5/7/84

NUMBER CTM-51
Appendix A
Page 2 of 2

SUBJECT:

CHEMICAL TEST METHOD FOR:
THERMOGRAVIMETRIC ANALYSIS

PREPARED BY: •

D. San Miguel

- 3.9 Turn recorder mode to % sample
- 3.10 Adjust Pen-1 until it reads 100 on the left hand of chart by turning the "% adjust knob"
- 3.11 Lower both pens, power knob - chart, push "heat" button on DSC-
- 3.12 Pen-2 should level off at 512 514°C mark, otherwise adjust upper limit and push heat until 512 - 514°C mark is attained on chart
- 3.13 When Pen-2 reaches 512 - 514°C, start timer for 20 minutes
- 3.14 Push "cool" button after 20 minutes, rais both pens, power knob -servo, and recorder mode - short

4.0 CALCULATION

- 4.1 Read % loss directly from chart (1% loss/small division on chart) using Pen-1

NOTE; Lower furnace only when temperature drops to 150°C or less



QUALITY ASSURANCE
TEST METHODS

DATE
5/7/84
Page 1 of 2

NUMBER
CTM-51
Appendix B

SUBJECT:
CHEMICAL TEST METHOD FOR:
THERMOGRAVIMETRIC ANALYSIS

PREPARED BY: •
D. San Miguel

1.0 MATERIAL: CCA-3 Fabric (1000°C Test)

2.0 EQUIPMENT: Setting

2.1 Heater Control: Load

Zero - + 20

Range - + 10

TGS - 2

2.2 Auto Balance:

Power - On

Filter - Lo

Tare - Pointer at the middle of green line

Recorder Range - 100

Recorder Mode - Short

2.3 Recorder:

Pen 2 (Zero pen by holding zero switch down; adjusting knob until pen is "0" on the right hand side of chart)

Range 1 volt

Polarity(-)

Pen 1 (Zero by following instruction of Pen 2)

Range No mv

Polarity(-)

Chart Speed 5 mm/min

2.4 Differential Scanning Calorimeter

Power On

Lower Unit 1000°C

Upper Limit 1000°C

Cooling Range 40°C/min

Heating Range 20°C/min

2.5 Air flow rate 80 ml/min (zero gas air)

3.0 TEST METHOD

3.1 Cut three 1/4" x 1/4" of fabric

3.2 Disengage spring latch of balance cover to release gas pressure

3.3 Loosen screw, then lower down furnace

3.4 Swing loading platform underneath platinum pan

3.5 Place samples on pan

3.6 Raise furnace carefully, tighten screw, and engage spring latch (Note; bubbles should appear on the outlet-tubing that is immersed in water if not, turn balance cover until bubbles come out)

3.7 Turn heater control to heat and recorder mode to Mg

3.8 Make mark on chart by lowering Pen-1 and advancing chart manually and raising pen again (reading will be 1 mg per small division, this will give the weight of sample)



QUALITY ASSURANCE
TEST METHODS

DATE 5/7/84
Page 2 of 2

NUMBER CTM-51
Appendix B

SUBJECT:

CHEMICAL TEST METHOD FOR:
THERMOVRAVIMETRIC ANALYSIS

PREPARED BY: •

D. San Miguel

- 3.9 Turn recorder mode to % sample
- 3.10 Adjust Pen-1 until it reads 100 on the left hand of chrt by turning the "% adjust knob"
- 3.11 Lower both pens, power knob - chart, push "heat" button on DSC
- 3.12 Pen-2 should level off at 1000°C mark, otherwise adjust upper limit and push heat until 1000°C mark is attained on chart
- 3.13 Push "cool" button after 1000°C is reached, raise both pens, power knob servo, and recorder mode - short

4.0 CALCULATION:

- 4.1 Read % loss directly from chart (1% loss/small division on chart) using Pen-1

NOTE: Lower furnace only when temperature drops to 150°C or less

THERMOGRAVIMETRIC ANALYSES

NASA 71108/71109 (resin, filler, fabric, prepreg) per CTM-51

Equipment

- Perkin-Elmer TGS-2
- Perkin-Elmer System 4

Sampling

- 5 - 10 mg in a platinum pan

Procedure

- Filter - Lo/med
- Recorder Mode - Short
- Recorder Range - 20 mv
- Heating Rate - 20 /min.
- Load Temperature - 50 C/min.
- Maximum Temperature - 950 C/hold for five minutes
- Chart Speed - 5 mm/min
- Atmosphere - Air
- Load sample in platinum pan; record weight
- Bring oven to load temperature to eliminate and recording of moisture loss
- Begin heating and recording until total weight loss is attained
- Add % weight loss vs. temperature increments
- Calibration for weight and temperature performed at start of project testing
- If pen becomes noisy - check air tank for good pressure
- Check to make sure sample and/or pan are not touching sides of oven
- Glass chamber might have build up causing static and needs cleaning



QUALITY ASSURANCE
TEST METHODS

DATE

8/1/84

NUMBER

CTM-53B
Page 1 OF 7

SUBJECT: CHEMICAL TEST METHOD FOR:
Atomic Absorption Determination of
Alkaline Impurities.

PREPARED BY: *

James B. Rock
James B. Rock
& Fred Bancroft

1.0 SCOPE

- 1.1 This procedure describes a method for determining alkaline metal impurities (sodium, potassium, calcium, lithium, and magnesium), in resins, fabrics and in preimpregnates.

2.0 EQUIPMENT AND MATERIALS

- 2.1 Atomic Absorption Spectrophotometer
 - 2.2 Analytical balance
 - 2.3 Volumetric flasks, 100 ml and 200 ml (one per sample and blank)
 - 2.4 Graduated cylinders, 10 ml & 50 ml
 - 2.5 Hot Plate
 - 2.6 Fume exhaust hood
 - 2.7 Timer
 - 2.8 Gloves; plastic
 - 2.9 Oven, Convection
 - 2.10 Muffle Furnace
 - 2.11 Platinum and ceramic crucibles (30 ml) one per sample
 - 2.12 Forceps
 - 2.13 Scissors
 - 2.14 Matrix standards (0.5 ppm for Na, K, Ca, Li & Mg)
 - 2.15 Nitric Acid, 6N
 - 2.16 Hydrochloric Acid 6N & 12N
 - 2.17 Hydrofluoric Acid 28N
 - 2.18 Cesium Chloride
 - 2.19 Potassium pyro-sulfate
 - 2.20 Deionized Water
- NOTE: Unless otherwise specified, all reagents are to be reagent grade or spectro grade, as applicable.

3.0 SAMPLING

- 3.1 Resins. Obtain representative 25 gm sample.
- 3.2 Fabrics. Obtain 16 sq. in.
- 3.3 Prepregs. Obtain 16 sq. in.

4.0 PROCEDURE

4.1 REAGENT PREPARATION

- 4.1.1 CsCl Stock Solution. Add about 1200 ml of deionized water to a two liter volumetric flask. Add 253g CsCl with agitation. After the CsCl is in solution, allow the solution to cool to ambient temperature and dilute to 2000 ml with deionized water to yield a 100,000 micrograms/ml solution.



QUALITY ASSURANCE
TEST METHODS

DATE

8-1-84

NUMBER

CTM-53B
Page 2 of 7

SUBJECT:

CHEMICAL TEST METHOD FOR:
Atomic Absorption Determination of
~~Alkaline Impurities~~

PREPARED BY:*

James B. Rock
& Fred Bancroft

4.2 CERAMIC CRUCIBLE PREPARATION

Ceramic crucibles are to be used for atomic absorption spectrophotometric testing of carbonaceous materials. All new crucibles are to be conditioned as below. Crucibles that have been in use for this procedure may be considered clean (proceed to par. 4.2.10).

- 4.2.1 Label the ceramic crucibles with an appropriate marking agent that will withstand at least 600°C.
- 4.2.2 Place crucibles on a hot plate in a laboratory fume hood and fill with 6N HCl. Using a medium hot plate setting, evaporate to near dryness then repeat procedure.
- 4.2.3 Remove crucibles from hot plate and allow to cool for five minutes.
- 4.2.4 Rinse the crucibles with deionized water at least three times.
- 4.2.5 Place the crucibles on the hot plate again and fill with 12N HCl. Using a medium hot plate setting, evaporate to near dryness and repeat.
- 4.2.6 Remove the crucibles from the hot plate and let stand for five minutes.
- 4.2.7 Rinse copiously with deionized water. Return to hot plate.
- 4.2.8 Fill the crucibles with 6N HCl and again evaporate to near dryness and repeat.
- 4.2.9 Remove the crucibles from the hot plate and allow to cool for five minutes.
- 4.2.10 Rinse the crucibles with deionized water copiously.
- 4.2.11 Place the crucibles in a muffle furnace, pre-conditioned to 600±15°C, for a minimum of three hours.
- 4.2.12 Remove the crucibles from the muffle furnace and desiccate until used.



QUALITY ASSURANCE
TEST METHODS

DATE

8/1/84

NUMBER

CTM-53B
PAGE 3 OF 7

SUBJECT:

CHEMICAL TEST METHOD FOR:
Atomic Absorption Determination of
Alkaline Impurities

PREPARED BY: *

James B. Rock
& Fred Bancroft

4.3 PLATINUM CRUCIBLE PREPARATION

Platinum crucibles are to be used for samples known to contain silica or for samples without a history of successful preparation in ceramic crucibles.

- 4.3.1 To clean the crucible, fuse about one gram of potassium pyrosulfate in the crucible. Rotate the crucible to distribute the melt along the inside walls.
- 4.3.2 Pour off the melt and allow the crucible to cool to ambient temperature.
- 4.3.3 Boil the crucibles in deionized water for about thirty (30) minutes. Rinse the boiled crucibles several times with deionized water.
- 4.3.4 Place the crucibles in a suitable container and fill with 6N HNO₃. Bring acid to a boil and maintain for one (1) hour. Pour off the hot acid and repeat.
- 4.3.5 Pour off the acid and rinse several times with deionized water.
- 4.3.6 Ignite the crucible in a muffle furnace (600 ± 15°C) for at least one hour. Pour off the hot acid and repeat.
- 4.3.7 Pour off the acid and rinse several times with deionized water.
- 4.3.8 Remove the crucibles from the muffle furnace and desiccate until used.

4.4 Sample Preparation

- 4.4.1 Using clean plastic gloves and clean scissors, cut the sample into approximate 1/2 inch squares and transfer into an analytically clean and labelled sample bottle. Unused sample will be retained. For multiple samples, every seventh crucible shall be a blank determination.
- 4.4.2 Assure that the crucibles have been conditioned per par. 4.2 or 4.3, as applicable.
- 4.4.3 Weigh and record the weight of the crucible to the nearest 0.1 mg.
- 4.4.4 Add approximately 2g of sample into the crucible using forceps. Weigh and record sample weight to nearest 0.1 mg.



QUALITY ASSURANCE
TEST METHODS

DATE

8/1/84

NUMBER

CTM-53B
Page 4 of 7

SUBJECT:

CHEMICAL TEST METHOD FOR: Atomic
Absorption Determination of Alkaline
Impurities.

PREPARED BY: •

James B. Rock
& Fred Bancroft

- 4.4.5 Transfer the crucibles to a convection oven and dry at $125 \pm 5^{\circ}\text{C}$ for two hours minimum.
- 4.4.6 Remove the crucibles from the oven and desiccate until ambient temperature is achieved.
- 4.4.7 Weigh and record the crucibles to the nearest 0.1 mg.
- 4.4.8 Transfer the crucibles to a muffle furnace and ash at $600 \pm 15^{\circ}\text{C}$ for 16 to 20 hours, or until constant weight is achieved.
- 4.4.9 Remove the crucibles from the muffle furnace. Desiccate until ambient temperature is achieved. Determine ash content, if required per par. 5.2.
- 4.4.10 Weigh and record the weights to the nearest 0.1 mg.
- 4.4.11 Place the crucibles on a hot plate and fill to about 75% of capacity with deionized water. Add 2 ml of 12N HCl to each crucible.
- 4.4.12 With the use of moderate heat, effect slow evaporation to near dryness. DO NOT ALLOW THE SAMPLES TO REACH DRYNESS. Repeat par. 4.11 and 4.12 as needed in order to effect solution. All crucibles must be treated identically.
- 4.4.13 Quantitatively transfer the solution from the crucibles into analytically clean volumetric flasks of appropriate size (to measure within the linear portion of the standard range).
- 4.4.14 Add sufficient CsCl stock solution to each volumetric flask to yield a 2000 ppm CsCl concentration when diluted to the mark.
- 4.4.15 Dilute each flask to the mark and agitate.
- 4.4.16 Agitate each solution prior to analysis.

4.5 PREPARATION OF STANDARD

- 4.5.1 Analysis of USP samples requires the use of a matrix standard below which the Beers Law relationship is linear. Empirical data has shown that for the Alkali metals and Alkaline earth metals, the calibration curve appears linear below 0.5 PPM. MATRIX: The matrix contain each of the following elements in deionized water Na, K, Ca, Li & Mg. The standard is made by adding (Volumetric pipet) 1 ml of a 1000 PPM stock solution for each of the elements into a 2000 ml volumetric flask and diluting to volume.



QUALITY ASSURANCE
TEST METHODS

DATE

8/1/84

NUMBER

CTM-53B
Page 5 Of 7

SUBJECT:

CHEMICAL TEST METHOD FOR: Atomic
Absorption Determination of Alkaline
Impurities.

PREPARED BY: •

James B. Rock
& Fred Bancroft

4.6 SPECTROPHOTOMETER START UP

4.6.1 Each laboratory shall generate an appropriate written procedure for instrument operation. The following is the U.S. Polymeric procedure.

4.6.1.1 Turn on air and acetylene valves.

4.6.1.2 Make sure air pressure is 40 psi and acetylene pressure is just below 15 psi.

4.6.1.3 Turn spectrophotometer switch on

4.6.1.4 Press oxidant button on left instrument panel and turn oxidant knob counter-clockwise until ball float goes down no further. This should be reading 12; if not, adjust knurled nut on nebulizer until a reading of 12 is obtained. Next turn oxidant knob clock-wise until a reading of 18 has been achieved.

4.6.1.5 Press fuel/oxidant button and adjust fuel knob until fuel ball float reads 7.

4.6.1.6 Remove nebulizer tube from D.I. H₂O and press flame on button to ignite flame.

4.6.1.7 Adjust fuel knob counter-clockwise until the yellow in the flame just disappears. (Make sure burner head height is 32).

4.7 ANALYSIS FOR POTASSIUM:

4.7.1 Adjust monochrometer to read 766.5 nm; place mode selector to a POSITION.

4.7.2 Place MA.count selector to the 1.0 position and turn the inner knob completely clockwise.

4.7.3 Aspirate the 0.5 PPM MATRIX Standard and adjust the monochrometer, in close vicinity of the originally set wave length, to obtain maximum deflection on the log scale meter (right instrument panel). This is fine tuning the wave length.

4.7.4 Aspirate deionized water or Nitric acid Digestion Blank and press auto zero button (you should at this time select the integration period depending on how stable you want the read out to be.)



QUALITY ASSURANCE
TEST METHODS

DATE

8/1/84

NUMBER

CTM-53B
PAGE 6 OF 7

SUBJECT:

CHEMICAL TEST METHOD FOR: Atomic
Absorption Determination of Alkaline
Impurities

PREPARED BY: *

James B. Rock
& Fred Bancroft

4.7.5 Start with a slit width of 80. Aspirate the 0.5 PPM MATRIX Standard and use the photomultiplier voltage knob and the inner MA/count knob to obtain a read out of 0.50 on the digital display. If this cannot be done, adjust slit selection either up or down depending on whether you want to increase or decrease the read, respectively.

4.7.6 Continuing to aspirate the 0.5 PPM Standard use the M/A count selector and inner knob to adjust the read out to 50 PPM for the 100 to 1 dilution.

4.7.7 Aspirate deionized water or nitric acid Digestion Blank and recheck zero.

4.7.8 Aspirate each sample. The read out will give direct PPM to be reported.

4.8 ANALYSIS FOR SODIUM, CALCIUM:

4.8.1 The procedure here is the same as for Potassium except that the initial monochrometer wave length for sodium is 589.0 nm. and for Calcium 422.7 nm. Calculate the curve for sodium that is linear between 0.1 and 2.5 micrograms per ml.

4.9 Analysis for Magnesium:

4.9.1 Adjust monochrometer to read 285.2 nm.

4.9.2 Place MA/count selector to the 1.0 position and turn the inner knob completely clock wise.

4.9.3 Place mode selector in A/DB position.

4.9.4 Turn lamp turret until Mg lamp locks into position; adjust knurled knob immediately above lamp on turret until lamp current reads 5 MA (right instrument panel)

4.9.5 Do Not aspirate standard, but fine tune wavelength by achieving maximum deflection on log scale by adjusting monochrometer.

4.9.6 Aspirate deionized water or nitric acid digestion blank and press auto zero button.

4.9.7 Aspirate 0.5 PPM MATRIX standard and set readout to 0.5 in same manner as for previous elements.

4.9.8 Adjust readout to 50 PPM with MA/count selector and inner knob as before with other elements.



QUALITY ASSURANCE
TEST METHODS

DATE

8/1/84

NUMBER

CTM-53B
PAGE 7 OF 7

SUBJECT: CHEMICAL TEST METHOD FOR: Atomic
Absorption Determination of Alkaline
Impurities

PREPARED BY: *

James B. Rock,
& Fred Bancroft

4.10 MISCELLANEOUS NOTES

4.10.1 The readout will be in direct sample parts per million (PPM) using this procedure.

4.10.2 The calibration (in terms of sample PPM) cannot be considered linear over 50 PPM. If a sample gives a read out of greater than 50 PPM, it must be diluted to give a readout within the operating range. The readout value must, in this case, be multiplied by this additional dilution to give the actual sample PPM.

4.10.3 Because dilute MATRIX standards deteriorate rapidly, a new 0.5 PPM standard must be prepared every two weeks to insure valid instrument calibration.

5.0 CALCULATIONS

5.1 Moisture.
$$\frac{(\text{raw sample weight}) - (\text{dry sample weight})}{(\text{raw sample weight})} \times 100 = \% \text{ moisture}$$

5.2 Ash
$$\frac{(\text{ash weight})}{(\text{dry sample weight})} \times 100 = \% \text{ ash}$$

5.3 Sodium, potassium, calcium, lithium or magnesium.
$$\frac{(\text{analyte } \mu\text{g/ml})}{(\text{sample g})} = \text{ppm sample}$$

6.0 REPORT

6.1 Moisture/content shall be reported to the nearest 0.10%. (when required)

6.2 Ash content shall be reported to the nearest 0.10% (when required)

6.3 Alkali metals contents shall be reported to the nearest 1.0 ppm.

CTM-53B

SPECIFICATION TITLE: Atomic Absorption Determination of Alkaline Impurities

DESCRIPTION OF CHANGE

Change paragraph 2.11 as follows:

WAS: Platinum and ceramic crucibles one per sample
NOW: Platinum crucibles one per sample

Change paragraph 2.14 as follows:

WAS: Matrix standards (0.5 ppm for Na, K, Ca, Li, Mg)
NOW: Matrix standards (0.25 ppm and 0.5 ppm for.....)

Change paragraph 2.18 as follows:

WAS: Cesium chloride
NOW: Lanthanum chloride

Change paragraph 4.1.1 as follows:

WAS: CsCl stock solution
NOW: To eliminate chemicals interferences between calcium and other ions, a Lanthanum chloride solution (5 g La Cl₃ dissolved in 2000 ml D. I. water) should be prepared and added to both CA standard solutions (0.25 ppm and 0.5 ppm) and analyzed samples.

Eliminate paragraph 4.2 entirely because the method does not indicate the use of ceramic crucibles.

Change paragraph 4.4.5 as follows:

WAS: Transfer the crucibles to a convection oven and dry at 125 ± 5 C for two hours minimum.
NOW: Transfer the crucibles with fabrics, prepreg or fillers to a convection oven and dry at 125 ± 5 C for two hours. If working with resins, transfer the crucibles to a convection oven and dry at 163 ± 3 C for two hours.

Change paragraphs 4.4.11-12 by replacing them as follows:

NOW: Place the crucibles on a hot plate and add a few drops of HF to each of them. Allow the solution to evaporate with the use of moderate heat. When the crucibles are dry, cool down slowly and fill them

with 2 ml HCl conc. and D. I. water to about 75% of capacity. In order to dissolve the salts, keep the crucibles on the hot plate at low-medium heat for about thirty minutes.

Change paragraph 4.4.13 as follows:

WAS: Quantitatively transfer the solution from the crucibles into analytically.....
NOW: After cooling down at room temperature, transfer the solutions from the crucibles into analytically volumetric flask of 200 ml capacity.

Change paragraph 4.4.14 as follows:

WAS: Add difficient CsCl stock solution.....
NOW: Add 1 ml of Lanthanum chloride solution (~1250 ppm) to each volumetric flask of 200 ml.

Change paragraph 4.5.1 as follows:

WAS: The standard is made by adding 1 ml of a 1000 ppm stock solution.....
NOW: Two different standard solutions should be prepared for each element, a 0.25 ppm and a 0.50 ppm, by adding respectively 0.5 ml and 1 ml of a 1000 ppm stock std. solution for each element into a 2000 ml volumetric flask and diluting to volume.

Change paragraph 4.6.1.5 as follows:

WAS: fuel ball float reads 7
NOW: fuel ball float reads 4 - 5

Change all of paragraph 4.7 as follows:

4.7 Analysis for Potassium and Lithium

4.7.1 Set the Video 12 AA/AE Spectrophotometer all the standard conditions for K and Lithium determination by flame emission.

- aspiration rate 1 ml/10 - 15 sec
- adjust monochromator to read 766.5 nm for K and 670.8 nm for Li
- bandwidth 1.00 nm for K and 0.5 nm for Li
- aspirate the 0.5 ppm standard solution and adjust the HV (high voltage) control until the log intensity meter reads in the green portion of the energy scale.

4.7.2 Aspirate D.I. water until the two standard solutions .025 ppm and 0.5 ppm construct the calibration curve for potassium, and store it. When press A/Z, aspirate the D.I. water and the computer will set the absorbance zero. When press A/C, aspirate std. solution 0.25 ppm and the computer will initiate the automatic calibrating routine. Aspirate each sample and the read out will give direct ppm to be reported.

Change paragraph 4.8 - 4.9 as follows:

NOW: 4.8 Analysis for sodium, calcium, magnesium

4.8.1 The procedure here is the same as for potassium, except that for sodium, magnesium and calcium, the determination is done by flame atomization, using dioxide filling, air/acetylene flame, and an adequate HCl lamp for each element.

4.8.1.1 The parameters for Na are:

- Current intensity 8 ma
- Adjust monochrometer to read X = 589.6 nm
- Band Width 1.00 nm

The lamp intensity meter should read in the green portion of the energy scale.

4.8.1.2 The parameters for calcium are:

- Current intensity 7 ma
- Adjust monochrometer to read X = 422.7 nm
- Band Width 1.00 nm

4.8.1.3 The parameters for Magnesium are:

- Current intensity 3 ma
- Adjust monochrometer to read X = 285.2 nm
- Band Width 1.00 nm

Complete paragraph 4.10.3 as follows:

NOW: Because dilute matrix standards deteriorate rapidly, new 0.25 ppm and 0.50 ppm standards must

Change paragraph 5.1 as follows:

$$\text{WAS: Moisture} = \frac{(\text{Raw Sample Weight}) - (\text{Dry Sample Weight})}{(\text{raw sample weight})}$$

$$\text{NOW: Moisture \%} = 100 - \frac{(\text{Dry Sample Weight} \times 100)}{(\text{Raw Sample Weight})}$$

Change paragraph 5.2 as follows:

$$\text{WAS: Ash \%} = \frac{\text{Ash Weight}}{\text{Dry Sample Weight}} \times 100$$

$$\text{NOW: Ash \%} = \frac{\text{Ash Weight}}{\text{Raw Sample Weight}} \times 100$$



U.S. Polymer, Inc.

QUALITY ASSURANCE
TEST METHODS

DATE

4/9/82

NUMBER

CTM-55

Page 1 of 10

SUBJECT:

CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY:

James B. Rock
James B. Rock

1.0 SCOPE

- 1.1 This method describes a procedure for routinely checking purity of solvents through the use of gas chromatography. It is similar to ASTM-D3792.

2.0 EQUIPMENT AND MATERIALS

2.1 Equipment

- a. Gas Chromatograph with Thermal Conductivity Detector-Hewlett-Packard Model 810 or any other with similar capability and performance (preferably equipped with an integrator).
- b. Two 6 feet by 1/8 inch stainless steel columns with packing of Porapak Q. 80/100 mesh. (Waters Associates)
- c. Hamilton syringe 701N or equivalent (10 microliter capacity)
- d. Pipettes, 10.0 ml, 1.0 ml, .1 ml
- e. Air tight sample bottles

2.2 Materials

- a. High Purity Solvents - Pure samples of each type of solvent to be checked as well as any suspected impurity solvents should be obtained. Nanograde purity is good. For acetone, Fisher A-20 is recommended.
- b. Distilled Water

3.0 SAMPLING

- 3.1 Representative Sample The sample of solvent to be injected into the G.C. should be representative. Exercise due care in certain cases where the test solvent has a constantly changing make-up.
- 3.2 Number of Determinations When quantifying solvent impurities, make at least 2 runs with the test samples to check accuracy.



QUALITY ASSURANCE
TEST METHODS

DATE 4/9/82

NUMBER CTM-55
Page 2 of 10

SUBJECT: CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY: •
James B. Rock

4.0 PREPARATION

4.1 Column Conditioning

- 4.1.1 If columns are new they should be conditioned prior to use according to manufacturer's instructions.
- 4.1.2 Prior to sample injection, the column should be clear of any contaminants from any previous injections. An isothermal run of about 225° for 1/2 hour will clear the column of most contaminants. Care should be exercised that the column temperature does not exceed the maximum rated temperature (250°C) since the column packing Porapak Q will start to degrade. This purging of the column should be done either with the columns disconnected from the thermal conductivity detectors, (Bridge current, off) or with the T.C. temperature well above the column temperature if the columns are connected. This will insure that material coming out of the column will not condense on a cooler T.C. filament. All other recommended precautions and procedures concerning column care contained in the G.C. operating manual should be followed.

- 4.2 Reference Sample Preparation Various reference samples of pure solvents as well as samples containing known percentages of "impurity" solvents should be made up and stored in air tight bottles. These reference samples will be run later to produce Chromatograms for comparison to test solvents. The analysis at hand will dictate what "impurity" solvents to use in the reference sample and in what percentages. For example, if the quality of the recycled acetone obtained from distilling common pre-preg systems slush is to be checked, the main impurities might be methyl cellosolve, toluene, methanol, and maybe some water. Therefore, reference samples of pure acetone and 1%, 2%, 3%, 5%, and 10% by volume of these impurities in acetone would probably be desirable.

5.0 PROCEDURE



QUALITY ASSURANCE
TEST METHODS

DATE 4/9/82

NUMBER CTM-55
Page 3 of 10

SUBJECT: CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY:
James B. Rock

5.1 Run Conditions Although run conditions will vary with the particular analysis, the conditions listed below will be close to those used for common low boiling solvent analysis (acetone, etc). If no idea of contaminate nature or percentages is known, a temperature program from 50°-195° is a good place to start.

- a. Helium Flow - Pre-set, equal on "A" and "B" columns, 30-40 ml/min.
- b. Main power, oven power, recorder power, chart drive, oven control, bridge switch, detector temp. switch - all on.
- c. Program Selector - Isothermal
- d. Oven Cover - Automatic
- e. Bridge Current - 200 milliamps
- f. Detector (T.C.) Temperature- 200°c
- g. Injector Temperature - 195°c
- h. Oven Temperature - 150°c
- i. Chart Speed - 1.0 inch/min.
- j. Attenuation - 16 (initially)

5.2 Sample Injection The integrator and recorder baselines will be quiet and without drift before each injection.

When both baselines are stable, 10.0 ml of air is injected into column "A". The chart is immediately marked at the injection point. This injection point to the maximum point of the air peak in minutes is T_m (retention time for unabsorbed gas).

After baseline has stabilized, a reference sample (2.0 ml) can then be injected. Again mark injection point and observe retention time (Tr') for each component of reference sample. The adjusted retention time (Tr') for each component is ($Tr' - T_{m_1}$).

After again allowing the baseline to stabilize, the reference sample injections are followed by the test samples. Many different reference samples may then be needed to make an adequate qualitative and quantitative analysis.



SUBJECT: CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY: •
James B. Rock

6.0 COMPOUND IDENTIFICATION

- 6.1 Compound identification is based upon the adjusted retention time (T_r'). The adjusted retention time is a function of the carrier gas, column temperature, column pressure drop, and other factors, thereby indicating the importance of maintaining constant conditions while comparing reference samples to test samples.
- 6.2 The recommended procedure for qualitative analysis is as follows: With the column at operating conditions, inject suitably sized samples of liquids, one compound at a time. Adjust the instrument attenuation so that the peak height is at least 50 percent of full scale. Inject the sample quickly into column "A". Again mark the injection point, and measure retention time (T_r') for each peak in minutes to at least 2 significant figures. Make duplicate determinations and use the average value compounds relative to at least one injected standard reference material. All data should be recorded carefully with each run. For qualitative analysis, the retention times of the sample peaks must then be matched with those of known standards obtained under the same operating conditions. Occasionally, it is necessary to analyze a sample in 2 different columns under different conditions, measuring the retention times of the components and compare them with previously determined standards, in order to determine which of 2 (or more) compounds having the same retention time is present. With common solvents, one type of column like Porapak Q. is often all that is necessary.

7.0 QUANTITATIVE ANALYSIS

- 7.1 The areas under the peaks of chromatogram are quantitative measures of the amounts of the corresponding compounds. The relative area is usually proportional to the concentration if the relative responses of the sample components are equal. If this is not the case, the corrected area of the component is used. It is obtained by dividing the peak area by the relative response of that component. Therefore, best results require a calibration curve of peak area, (or height) versus known amounts of pure components for the given operating conditions.
- 7.2 Although the establishment of the true peak area by integration is the standard method for quantitative analysis, multiplying the peak by the width of the peak taken at the half-height gives proportional



SUBJECT: CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY: *
James B. Rock

7.2 (Continued)

values especially for sharp, symmetrical, completely resolved peaks. With this method, a chart speed that will give at least a 5 mm half-width should be used.

7.3 When the volumes injected are the same, the following Relationships apply:

$$V_1 = (H_1/H_2) \times V_2$$

$$\text{and } W = V_1 \times (D_1/D_2)$$

Where:

V_1 = Vol. percent of sample component

H_1 = Compound peak area or height in sample

H_2 = Compound peak area or height in standard

V_2 = Volume percent of sample component in standard

W = Weight percent of sample component

D_1 = Density of pure sample component

D_2 = Density of sample

- 7.4 An outline of the recommended procedure used for the quantitative calibration is as follows: With the column at equilibrium operating conditions, inject liquid samples of pure components, one compound at a time. More than one at a time is acceptable if you're sure what peak corresponds to which compound. Vary the sample size (or concentration) to cover the desired range. Samples of a given size should be injected until at least three peaks have the same area, ideally ± 1 percent at the same attenuation. Adjust the attenuation in all cases to keep the peak on scale and with a height of at least 50 percent. Data should be converted to some attenuation previously chosen as a basis for calculations, for example, $\times 1$, $\times 8$, $\times 64$, etc. Multiply the actual peak height by its corresponding attenuation and divide the basis attenuation. Plot this peak height versus quantity of each compound and connect the data points with a smooth calibration curve. Label the axes on the plot and for each such plot show the following information: Date, column description, and operating conditions. Record also the purity of the compounds used. The syringe, pipette, or other device must be flushed



QUALITY ASSURANCE
TEST METHOD

DATE 4/9/82

NUMBER CTM-55
Page 6 of 10

SUBJECT: CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using GAS Chromatography

PREPARED BY: *
James B. Rock

7.4 (Continued)

at least 3 times with each new compound prior to
injecting a sample of that component.

Actual sample injections will generate peaks which
can then be easily quantified using the calibration
charts.

7.5 An example of a possible analysis follows:

All runs are made with identical conditions.

All samples injected are 2.0 ml.

Reference samples -Tr'

Acetone - 7.5 min.

Methanol - 2.7 min.

Ethanol - 5.0 min.

From Calibration Curves

<u>Attenuation</u>	<u>Peak Area</u>	<u>Volume Percent of Ethanol</u>
16	25	1
16	50	2
16	100	4
8	100	8

Sample peak of impurity in solution is Tr' = 5.0
min. and peak area of 75 at 16 attenuation. From
previous calibration work, peak is ethanol. 3% by
Volume.

8.0 CALIBRATION

8.1 Before each calibration and series of determinations
(or daily) condition the column at 200°C for 1 h
with carrier gas flow.

8.2 Determination of Relative Response Factors - Anhydrous
2-propanol is used as an internal standard. The
response factor to water relative to the standard
is determined by means of the following procedure.
See Fig. 1 for a typical chromatogram. It is good
practice to determine the response factor daily
or with each series of determinations.

8.2.1 Weigh about 0.2g of water and 0.2 g of 2-propanol
to the nearest 0.1 mg into a septum sample vial.



SUBJECT:

CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY: •

James B. Rock

8.2.1 (Continued)

If it has been determined that a correction for the water content is necessary, weigh 2 ml of dimethylformamide into the vial. If the dimethylformamide is anhydrous, simply add 2 ml of it as weighing is not necessary.

8.2.2 Inject a 1 μ l aliquot of the above solution onto the column and record the chromatogram. The retention order and approximate retention times after the air peak are 1. water, about 0.7 min; 2. 2-propanol, about 2.8 min, and 3. DMF, about 7 min.

8.2.3 The preferred procedure to obtain the water content of the DMF is by Karl Fischer titration. If this has been done, calculate the response factor for water by means of the following equation:

$$R = \frac{W_i A_{H_2O}}{(W_{H_2O} + P W_s) A_i}$$

where:

R = response factor

W_i = weight of 2-propanol

W_{H₂O} = weight of water added

W = weight of dimethyl-formamide

A_{H₂O} = area of water peak

A_i = area of 2-propanol peak and

P = $\frac{\text{weight\% water in DMF}}{100}$

8.2.4 If Karl Fischer titration is not available, the following procedure may be used to obtain a reasonable estimate of the response factor.

8.2.4.1 Inject the same size aliquot of DMF and 2-propanol mixture, but without added water, as a blank, Note the area of the water peak in the blank.

8.2.4.2 The response factor for water is calculated by means of the following equation:

$$R = \frac{W_i (A_{H_2O} - B)}{W_{H_2O} A_i}$$

Where:

R = response factor

W_i = weight of 2-propanol

W_{H₂O} = weight of the water



QUALITY ASSURANCE
TEST METHOD

DATE 4/9/82

NUMBER CTM-55
Page 8 of 10

SUBJECT:
CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY:
James B. Rock

A_i = area of propanol peak
 A_{H_2O} = area of the water peak
 B = area of the water peak the blank

9.0 PROCEDURE

- 9.1 Weigh to the nearest 0.1 mg 0.6 g of water-reducible sample and 0.2 g of 2 propanol into a septum vial. Add 2 ml of dimethylformamide into the vial. Seal the vial. Prepare a blank containing the 2 propanol and dimethylformamide but no sample.
- 9.2 Shake the vials on a wrist action shaker or other suitable device for 15 min. To facilitate settling of solids allow the samples to stand for 5 min. just prior to injection into the chromatograph. Low speed centrifugation may also be used.
- 9.3 Inject a 1 μ l sample of the supernatant from the prepared solutions onto the chromatographic column. Record the chromatograms using the conditions described in Table 1.

10.0 CALCULATIONS

- 10.1 Measure the area of the water peak and the 2 propanol internal standard peak and multiply each area by the appropriate attenuation factor to express the peak areas on a common basis. Use of an electronic integrator is recommended to obtain the best accuracy and precision. However, triangulation, planimeter, paper cut out, or ball and disk integrator may be used.
- 10.2 Determine the water concentration in the sample by means of the following equation:

$$H_2O, \% = \frac{A_{H_2O} \times W_i \times 100}{A_i \times W_p \times R}$$

Where:

A_{H_2O} = area of water peak
 A_i = area of 2 propanol peak
 W_i = weight of 2 propanol added
 W_p = weight of sample and
 R = response factor determined in 8.2

SUBJECT: CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas ChromatographyPREPARED BY: •
James B. Rock

10.3 Correction for Water Content of Solvent

10.3.1 If the blank indicates the presence of a detectable peak for water in the dimethylformamide used as solvent, make a correction in the calculation.

10.3.2 To make the correction, the water content of the dimethylformamide is determined either by chromatography (8.2.4) or, preferably, by Karl Fischer titration (8.2). Calculate the water content due to solvent by using the following equation:

$$H_2O(S), \% = \frac{(W_s)(P) 100}{W_p}$$

where:

W = weight of dimethylformamide

 W^S = weight of sample, and $P^D = \frac{\text{weight \% water in DMF}}{100}$

10.3.3 The water content of the sample in this case is the difference between the total percent determined in 10.2 and the correction for the solvent water content as determined in 10.3.2.

TABLE I Instrument Parameters (Typical Conditions)

Detector	thermal conductivity 1.22m by 3.2
Column	mm outside diameter packed with 60 to 80 mesh porous polymer packing
Temperatures:	
Sample inlet	200°C
Detector	240°C
Column	
Initial	80°C
Final	170°C
Program rate	30°C/min
Carrier Gas	helium or nitrogen
Flow rate	50 ml/min
Detector current	150 mA
Specimen size	1µl

For isothermal operation set the column temperature at 140°C. After the 2 propanol has cleared the column adjust the temperature to 170°C until DMF clears the column. Reset the temperature to 140°C for subsequent runs.



QUALITY ASSURANCE
TEST METHOD

DATE 4/9/82

NUMBER CTM-55
Page 10 of 10

SUBJECT: CHEMICAL TEST METHOD FOR:
Quality Control of Solvents
Using Gas Chromatography

PREPARED BY: *
James B. Rock

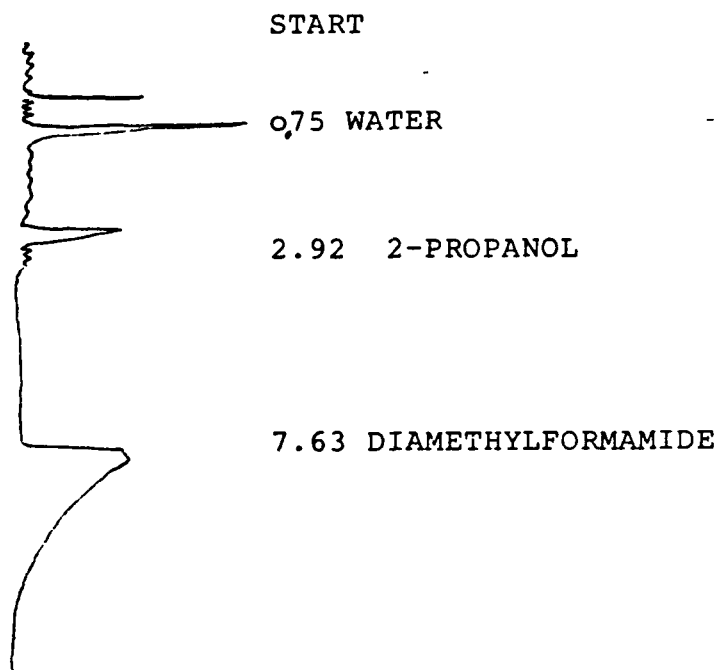


FIG. 1 Typical Chromatogram.



Quality Assurance
Test Methods

DATE

9-22-87

NUMBER CTM-55

Addendum A

Page 1 of 4

SUBJECT:

Chemical Test Method For: Solvent Identification in Phenolics

PREPARED BY:

John N. Zocco J. Zocco
APPROVED BY: Hamid M. Quraishi

1.0 SCOPE

This method describes a procedure for identifying a variety of solvents in phenolics through the use of gas chromatography.

2.0 EQUIPMENT AND MATERIALS

- 2.1 Perkin-Elmer Sigma 300 FID/HWD Chromatograph
- 2.2 Perkin-Elmer 3600 Data Station
- 2.3 Perkin-Elmer Chromatographics Intelligent Terminal
- 2.4 Perkin-Elmer 660 Printer
- 2.5 Perkin-Elmer Chromatography Interface
- 2.6 Glass Column, 6 ft X 1/4 in. 0.1% AT-1000 on Graphpac, 80/100 mesh
- 2.7 Double-sided Double-density 5-1/4 inch Diskettes
- 2.8 Ten Microliter Gas-tight Syringe
- 2.9 Septa, 7/16 inch
- 2.10 Serum Bottles, 10-ml
- 2.11 Rubber Stoppers, 13 X 20-mm Red
- 2.12 Silicone Stoppers, 13 X 20-mm Amber
- 2.13 Hycar Stoppers, 13 X 20-mm Black
- 2.14 Tear-off Aluminum Seals, 20-mm
- 2.15 Fermpress
- 2.16 Disposable Pipettes, 10-ml
- 2.17 Tetrahydrofuran, High Purity
- 2.18 Methanol, High Purity
- 2.19 Ethanol, High Purity
- 2.20 Methylene Chloride, High Purity
- 2.21 Acetone, High Purity
- 2.22 Isopropanol, High Purity
- 2.23 Acetonitrile, High Purity
- 2.24 p-Cresol, High Purity
- 2.25 Methyl Ethyl Ketone, High Purity
- 2.26 Furfural, High Purity
- 2.27 Toluene, High Purity
- 2.28 Chlorobenzene, High Purity
- 2.29 Benzene, High Purity
- 2.30 Phenol, High Purity
- 2.31 2,3-Dimethylbutane, High Purity
- 2.32 2-Methylpentane, High Purity
- 2.33 3-Methylpentane, High Purity
- 2.34 n-Hexane, High Purity
- 2.35 Methyl Cyclopentane, High Purity
- 2.36 Helium, High Purity
- 2.37 Hydrogen, High Purity
- 2.38 Air, High Purity



Quality Assurance
Test Methods

DATE
9-22-87

NUMBER CTM-55
Addendum A
Page 2 of 4 *D*

SUBJECT:

Chemical Test Method For: Solvent Identification in Phenolics

PREPARED BY:

John D. Jones J. Zocco *JZ*
APPROVED BY: Hamid M. Quraishi *HMQ*

3.0 PREPARATION OF SAMPLES

- 3.1 All phenolic samples such as 91-LD, USP-39A, F-064 (P-28), and F-064 (P-33) are prepared in the same manner.
- 3.2 Weigh one gram of sample into a serum bottle.
- 3.3 Dispense 10 ml of tetrahydrofuran (THF) into serum bottle. Concentration of sample is 0.1 gm/ml.
- 3.4 Cap-off serum bottle with a silicone stopper and an aluminum seal. Close bottle tight with fermpress.
- 3.5 Shake sample for fifteen minutes.
- 3.6 Samples which contain graphite should be centrifuged for thirty minutes at maximum speed prior to sampling.

4.0 INSTRUMENT PROGRAMMING

4.1 Perkin-Elmer Sigma 300 FID/HWD Chromatograph

- 4.1.1 Detector is FID (flame ionization), Detector "A".
- 4.1.2 Hydrogen pressure is 20 psi.
- 4.1.3 Air pressure is 30 psi.
- 4.1.4 Carrier gas (Carrier Flow A) is helium
- 4.1.5 Helium flowrate is 30 ml/min with an inlet pressure of 60 psig.
- 4.1.6 Detector temperature is 220 C.
- 4.1.7 Injector temperature is 200 C.
- 4.1.8 The initial column temperature (Oven Temp. 1) is 60 C.
- 4.1.9 The final column temperature (Oven Temp. 2) is 210 C.
- 4.1.10 The heating rate (Rate 1) is 5 C/min.
- 4.1.11 The total run time is thirty minutes. Time 1 and Time 2 are both zero.
- 4.1.12 The recorder attenuator is 1.
- 4.1.13 The range is X1.
- 4.1.14 The zero reverse is +.
- 4.1.15 The recorder selector is 10 mV.

4.2 Perkin-Elmer 3600 Data Station

- 4.2.1 See Table 1. This is the method, "TEST", for running phenolics.
- 4.2.2 The number of runs depends on the amount of disk space that is left.
- 4.2.3 The disk file prefix is a five character term that is used to store the runs.

5.0 PROCEDURE

- 5.1 Prior to sample injection, the column must be conditioned according to CTM-55, section 4.1.



Quality Assurance
Test Methods

DATE

9-22-87

NUMBER CTM-55
Addendum A

Page 3 of 4 (12)

SUBJECT:

Chemical Test Method For: Solvent Identification in Phenolics

PREPARED BY:

John J. Zocco J. Zocco
APPROVED BY: Hamid M. Quraishi

- 5.2 In order to determine what the components in the phenolic samples are, a variety of pure solvents were run prior to running any samples, and their retention times are listed in Table 2.
- 5.3 The pure solvents run were methanol, ethanol, isopropanol, methylene chloride, acetone, methyl ethyl ketone, tetrahydrofuran, acetonitrile, p-cresol, phenol, furfural, toluene, chlorobenzene, benzene, 2,3-dimethylbutane, 2-methylpentane, 3-methylpentane, n-hexane, and methyl cyclopentane.
- 5.4 Place "Chromatographics 2 software, Rev. D" diskette in drive zero.
- 5.5 Place a formatted disk for storing data in drive one.
- 5.6 Turn computer on. Type in the time and date. When "PETOS>" appears, type "CHROM". Press return key.
- 5.7 When "CIT2>" appears, press the "METHOD" key on the keyboard. Type "TEST" for the method name. See Table 1. Press the "ACCEPT" key.
- 5.8 When "Modify Method Test [Y or N]" appears, type "Y". Page 1 will appear - see Table 1. Press the "ACCEPT" key.
- 5.9 Page 2 now appears. Type in the sample name, e.g. "TETRAHYDROFURAN". The starting number could be "1" as is in this case. Press the "ACCEPT" key.
- 5.10 Page 6 now appears. Pages 3 to 5 are not used in this method. Type a name for the disk file prefix, e.g. "JGZ01".
- 5.11 When "Overwrite Method File:-TEST [Y or N]?" appears, type "Y".
- 5.12 When "Initialise Interface 1 [Y/N]?" appears, type "Y". The sample is now ready to be injected.
- 5.13 Program the chromatograph - see section 4.1. Make sure that the air, helium, and hydrogen tanks are turned on. All inlet pressures should be set at 60 psig.
- 5.14 When the ready light on the chromatograph appears, ignite the detector.
- 5.15 Draw approximately 9.5 ul of air and 0.5 ul of sample into a 10-ul gas-tight syringe.
- 5.16 Insert needle of syringe into injector and push sample through. Press the ready light on chromatograph.
- 5.17 After the run has ended, a results table and plot of the chromatogram will be printed. The results table is shown in Table 3. The plot of the chromatogram is shown in Figure 1.
- 5.18 Make duplicate runs. Take the average retention times.

6.0 CALCULATIONS

- 6.1 See CTM-55, section 6.0.
- 6.2 Compound identification is based upon the adjusted retention time (Tr'). The adjusted retention time is a



Quality Assurance
Test Methods

DATE

9-22-87

NUMBER CTM-55

Addendum A

Page 4 of 4 *ES*

SUBJECT:

Chemical Test Method For: Solvent Identification in Phenolics

PREPARED BY:

John D. Zocco J. Zocco *ES* *HA*

APPROVED BY: Hamid M. Quraishi

function of the carrier gas, column temperature, column pressure drop, and other factors, thereby indicating the importance of maintaining constant conditions while comparing reference samples to test samples.

6.3 The retention times of the sample peaks must then be matched with those of known standards obtained under the same operating conditions. See Table 2.

7.0 CONCLUSION

By following the method above, excellent reproducibility is obtained in identifying many different solvents present in phenolics.

TABLE 1.

METHOD "TEST"

***** METHOD TEST *****

.1% AT1000 GRAPHFAC

DATA ACQUISITION PARAMETERS	
INTERFACE NUMBER	1
DATA CHANNEL	A
DATA START TIME IN MINS.	0.0
DATA END TIME IN MINS.	30.0
PEAK DETECTION THRESHOLD	2.00
MINIMUM PEAK WIDTH IN SECS.	15.0
DISPLAY AND STORE DATA DURING RUN	Y
TIMED EVENTS TABLE REQUIRED	N
MORE DATA PROCESSING PAGES	N
CALCULATION PARAMETERS	
CALIBRATION BY AREA/HEIGHT	A
SKIM FACTOR	5
CONCENTRATION UNITS	
REPORT GENERATION	
AREA/HEIGHT PERCENT	Y
NORMALISED PERCENT	N
EXTERNAL STANDARD	N
INTERNAL STANDARD	N
GROUPED REPORT	N
RETENTION TIME WINDOWS	
PERCENT	5
FIXED	5
SAVE DATA	
PLOT FILES	Y
REPORT FILES	Y
PRINT PLOT	Y
NUMBER OF RUNS	1
CHANNEL A	
SCREEN FULL SCALE MV	1000
AREA REJECTION	0
DISC FILE PREFIX	JGZ01
PRINTED PLOT MIN/PAGE	30
VERTICAL SCALE FACTOR	1

TABLE 2.
G.C. SOLVENTS

GC SOLVENTS

January 14, 1967

GAS CHROMATOGRAPHY DATA INTERPRETATION

TEST METHOD CTM-55

SOLVENT/MONOMER	RETENTION TIME MINS.
MEOH	.6
ETHANOL	1.18
MECL2	1.28
ACETONE	1.45
IPA	1.83
THF	3.08
ACETONITRILE	3.2
CRESOL	4.03
MEK	4.08
FURFURAL	15.03
TOLUENE	17.98
CHLOROBENZENE	19.6
PHENOL	22.08

TABLE 3.

RESULTS

***** METHOD PARAMETERS *****

METHOD: TEST : .1% AT1000 GRAPH PAC
PLOT: JGZ01

SAMPLE: TETRAHYDROFURAN
INJECTION NUMBER: 01

RUN AT: 08:38 ON: 87-01-15 INTERFACE NO: 1A

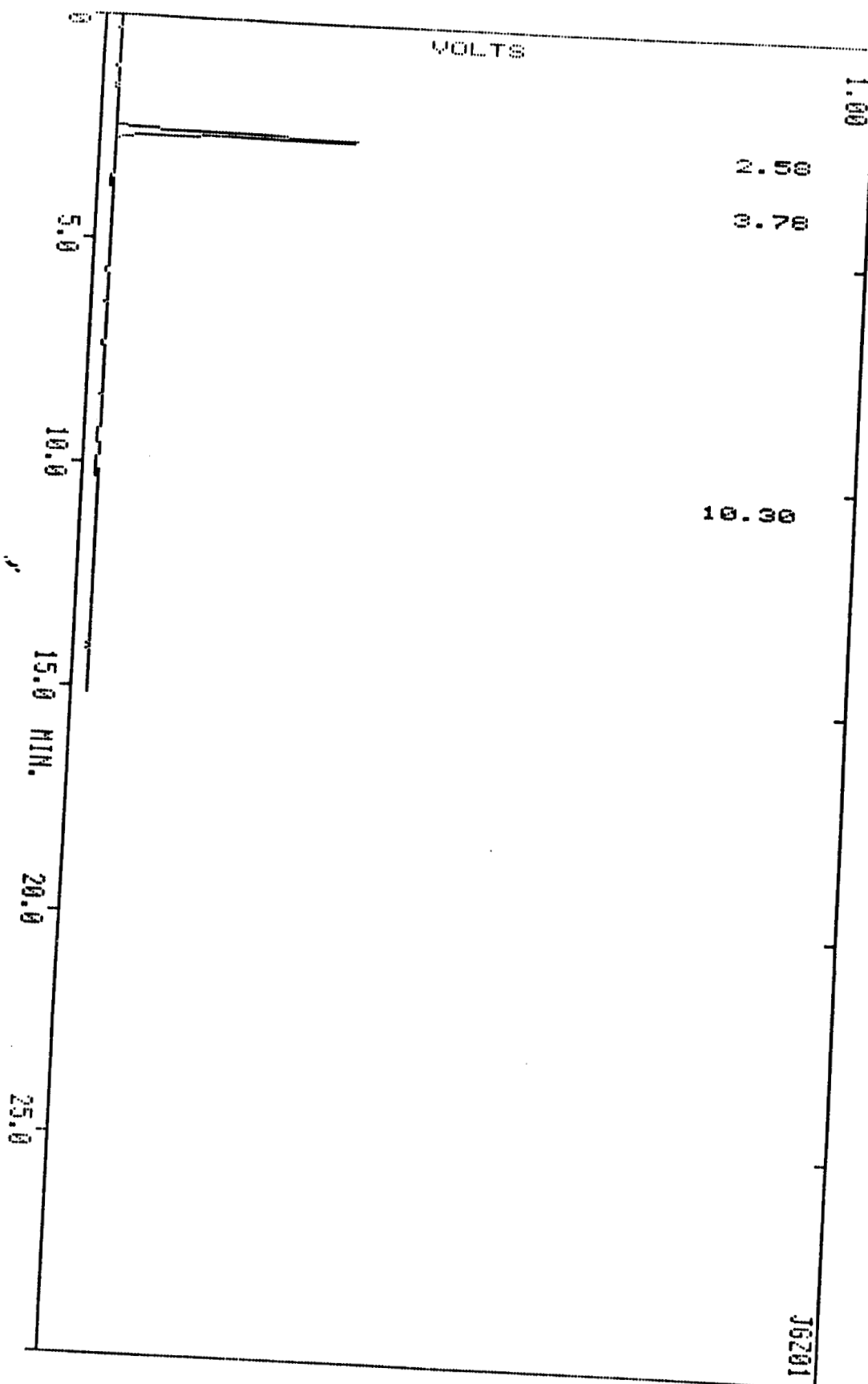
START TIME : 0.0
END TIME : 15.2
DETECTION THRESHOLD : 2.00
MINIMUM PEAK WIDTH : 15.0
AREA REJECT THRESHOLD: 0.0

***** AREA/HEIGHT PERCENT *****

PK NO	RETEN TIME	B C	PEAK AREA	AREA %	PEAK HEIGHT	HEIGHT %
1	2.57	1	19552	98.649	3304	99.310
2	3.77	1	112	0.564	11	0.329
3	10.30	1	156	0.787	12	0.361

3 PEAKS; TOTAL AREA: 19820
HEIGHT: 3327

Figure 1.
G.C. CHROMATOGRAM





QUALITY ASSURANCE
TEST METHODS

DATE
SEPT. 11, 1987

NUMBER CTM-55
ADDENDUM B
PAGE 1 OF 4

SUBJECT:

CHEMICAL TEST METHOD FOR:
PHENOL BY GAS CHROMATOGRAPHY

PREPARED BY:

J. ZOCCO *[Signature]*
APPROVED BY: Hamid M. Quraishi *[Signature]*

ADDENDUM TO CTM-55

1.0 SCOPE

To test resinous materials for % Wt./Wt. Phenol by Gas Chromatography.

2.0 EQUIPMENT AND MATERIALS

- 2.1 Perkin-Elmer Sigma 300 FID/HWD Chromatograph
- 2.2 Perkin-Elmer 3600 Data Station
- 2.3 Perkin-Elmer Chromatographics Intelligent Terminal
- 2.4 Perkin-Elmer 660 Printer
- 2.5 Perkin-Elmer Chromatography Interface
- 2.6 Glass Column, 1 meter x 2 mm, Tenox GC, 60/80 Mesh
- 2.7 Double-sided Double-density 5-1/4 inch Diskettes
- 2.8 Ten Microliter Gas-tight Syringe
- 2.9 Septa, 7/16 inch
- 2.10 Serum Bottles, 10-ml
- 2.11 Rubber Stoppers, 13 X 20-mm Red
- 2.12 Silicone Stoppers, 13 X 20-mm Amber
- 2.13 Hycar Stoppers, 13 X 20-mm Black
- 2.14 Tear-off Aluminum Seals, 20-mm
- 2.15 Fermpress
- 2.16 Disposable Pipettes, 10-ml
- 2.17 Tetrahydrofuran, High Purity
- 2.18 Methanol, High Purity
- 2.19 Ethanol, High Purity
- 2.20 Methylene Chloride, High Purity
- 2.21 Acetone, High Purity
- 2.22 Isopropanol, High Purity
- 2.23 Acetonitrile, High Purity
- 2.24 p-Cresol, High Purity
- 2.25 Methyl Ethyl Ketone, High Purity
- 2.26 Furfural, High Purity
- 2.27 Toluene, High Purity
- 2.28 Chlorobenzene, High Purity
- 2.29 Benzene, High Purity
- 2.30 Phenol, High Purity
- 2.31 2,3-Dimethylbutane, High Purity
- 2.32 2-Methylpentane, High Purity
- 2.33 3-Methylpentane, High Purity
- 2.34 n-Hexane, High Purity
- 2.35 Methyl Cyclopentane, High Purity
- 2.36 Helium, High Purity
- 2.37 Hydrogen, High Purity
- 2.38 Air, High Purity



QUALITY ASSURANCE
TEST METHODS

DATE
SEPT. 11, 1987

NUMBER CTM-55
ADDENDUM B
PAGE 2 OF 4

SUBJECT: CHEMICAL TEST METHOD FOR:
PHENOL BY GAS CHROMATOGRAPHY

PREPARED BY: J. ZOCCO
John N. Zocco
APPROVED BY: Hamid M. Quraishi

3.0 PREPARATION OF SAMPLES

- 3.1 All phenolic samples such as 91-LD, USP-39A, F-064 (P-28), and F-064 (P-33) are prepared in the same manner.
- 3.2 Weigh 10.0 ± 0.2 grams of sample into a 100 ml volumetric flask.
- 3.3 Dispense 2-propanol (isopropyl alcohol), ACS Grade, to volume.
- 3.4 Mix at room temperature until dissolved.
- 3.5 For resin mix samples which contain carbon black, samples shall be filtered by 0.2 micron PTFE filter prior to injection.

4.0 INSTRUMENT PROGRAMMING

4.1 Perkin-Elmer Sigma 300 FID/HWD Chromatograph

- 4.1.1 Detector is FID (flame ionization), Detector "A".
- 4.1.2 Hydrogen pressure is 20 psi.
- 4.1.3 Air pressure is 30 psi.
- 4.1.4 Carrier gas (Carrier Flow A) is helium
- 4.1.5 Helium flowrate is 30 ml/min with an inlet pressure of 60 psig.
- 4.1.6 Detector temperature is 320 C.
- 4.1.7 Injector temperature is 250 C.
- 4.1.8 The initial column temperature (Oven Temp. 1) is 70 C.
- 4.1.9 The final column temperature (Oven Temp. 2) is 320 C.
- 4.1.10 The heating rate (Rate 1) is 8 C/min.
- 4.1.11 The total run time is thirty minutes.
- 4.1.12 The recorder attenuator is 1.
- 4.1.13 The range is X1.
- 4.1.14 The zero reverse is +.
- 4.1.15 The recorder selector is 10 mV.

4.2 Perkin-Elmer 3600 Data Station

- 4.2.1 See Table 1. This is the method, "TEST", for running phenolics.
- 4.2.2 The number of runs depends on the amount of disk space that is left.
- 4.2.3 The disk file prefix is a five character term that is used to store the runs.

5.0 PROCEDURE

- 5.1 Prior to sample injection, the column must be conditioned according to CTM-55, section 4.1.



QUALITY ASSURANCE
TEST METHODS

DATE
SEPT. 11, 1987

NUMBER CTM-55
ADDENDUM B
PAGE 3 OF 4

SUBJECT: CHEMICAL TEST METHOD FOR:
PHENOL BY GAS CHROMATOGRAPHY

PREPARED BY: J. ZOCCO *J. Zocco*
APPROVED BY: Hamid M. Quraishi *H. M. Quraishi*

- 5.2 Place "Chromatographics 2 software, Rev. D" diskette in drive zero.
- 5.3 Place a formatted disk for storing data in drive one.
- 5.4 Turn computer on. Type in the time and date. When "PETOS>" appears, type "CHROM". Press return key.
- 5.5 When "CIT2>" appears, press the "METHOD" key on the keyboard. Type "TEST" for the method name. See Table 1. Press the "ACCEPT" key.
- 5.6 When "Modify Method Test [Y or N]" appears, type "Y". Page 1 will appear - see Table 1. Press the "ACCEPT" key.
- 5.7 Page 2 now appears. Type in the sample name, e.g. "TETRAHYDROFURAN". The starting number could be "1" as is in this case. Press the "ACCEPT" key.
- 5.8 Page 6 now appears. Pages 3 to 5 are not used in this method. Type a name for the disk file prefix, e.g. "JGZ01".
- 5.9 When "Overwrite Method File:-TEST [Y or N]?" appears, type "Y".
- 5.10 When "Initialise Interface 1 [Y/N]?" appears, type "Y". The sample is now ready to be injected.
- 5.11 Program the chromatograph - see section 4.1. Make sure that the air, helium, and hydrogen tanks are turned on. All inlet pressures should be set at 60 psig.
- 5.12 When the ready light on the chromatograph appears, ignite the detector.
- 5.13 Draw approximately 9.0 ul of air and 1.0 ul of sample into a 10-ul gas-tight syringe.
- 5.14 Insert needle of syringe into injector and push sample through. Press the ready light on chromatograph.
- 5.15 After the run has ended, a results table and plot of the chromatogram will be printed. The results table is shown in Table 3. The plot of the chromatogram is shown in Figure 1.
- 5.16 Make duplicate runs. Take the average retention times.

6.0 CALCULATIONS

- 6.1 See CTM-55, section 6.0.
- 6.2 Compound identification is based upon the adjusted retention time (T_r'). The adjusted retention time is a function of the carrier gas, column temperature, column pressure drop, and other factors, thereby indicating the importance of maintaining constant conditions while comparing reference samples to test samples.
- 6.3 The retention times of the sample peaks must then be matched with those of known standards obtained under the same operating conditions. See Table 2.
- 6.4 Using the average area of a 2.00% Phenol Standard input, the following:



QUALITY ASSURANCE
TEST METHODS

DATE
SEPT. 11, 1987

NUMBER CTM-55
ADDENDUM B
PAGE 4 OF 4

SUBJECT: CHEMICAL TEST METHOD FOR:
PHENOL BY GAS CHROMATOGRAPHY

PREPARED BY: J. ZOCCO *[Signature]*
APPROVED BY: Hamid M. Quraishi

Retention Time - 13 minutes
Concentration - % weight x 10⁴
Area - Average of two runs
Name Phenol

Note: The dilution of neat resin at 1:10 is corrected for by the use of a factor in the calculation of % Phenol

6.5 Record sample weight, % solids, area of chromatogram peak, and % Phenol.

7.0 CONCLUSION

By following the method above, excellent reproducibility is obtained in identifying many different solvents present in phenolics.

TABLE 1

ORIGINAL PAGE IS
OF POOR QUALITY

***** METHOD TEST *****

.1% AT1000 GRAPH PAC

DATA ACQUISITION PARAMETERS

INTERFACE NUMBER	1
DATA CHANNEL	B
DATA START TIME IN MINS.	0.0
DATA END TIME IN MINS.	20.0
PEAK DETECTION THRESHOLD	2.00
MINIMUM PEAK WIDTH IN SECS.	15.0

DISPLAY AND STORE DATA DURING RUN	Y
-----------------------------------	---

TIMED EVENTS TABLE REQUIRED	N
-----------------------------	---

MORE DATA PROCESSING PAGES	Y
----------------------------	---

CALCULATION PARAMETERS

CALIBRATION BY AREA/HEIGHT	B
SKIM FACTOR	5
CONCENTRATION UNITS	% MEK * 10

REPORT GENERATION

AREA/HEIGHT PERCENT	Y
NORMALISED PERCENT	N
EXTERNAL STANDARD	Y
INTERNAL STANDARD	N
GROUPED REPORT	N

RETENTION TIME WINDOWS

PERCENT	5
FIXED	5

PEAK NO	RETEN TIME	RESPONSE FACTOR	PEAK NAME	GROUP
1	13.00	8.0965	MEK	0

SAVE DATA

PLOT FILES	Y
REPORT FILES	Y
PRINT PLOT	Y

DILUTION FACTOR	1.0000
SAMPLE VOLUME	1.0000
STANDARD VOLUME	1.0000

NUMBER OF RUNS	22
----------------	----

CHANNEL B

SCREEN FULL SCALE MV	100
AREA REJECTION	0
DISC FILE PREFIX	DAT01
PRINTED PLOT MIN/PAGE	20
VERTICAL SCALE FACTOR	1

TABLE 2

ORIGINAL PAGE IS
OF POOR QUALITY

***** METHOD PARAMETERS *****

METHOD: TEST : .1% AT1000 GRAPH PAC
PLOT: DAT34

SAMPLE: 10 % MEK IN MECL2
INJECTION NUMBER: 34

RUN AT: 10:19 ON: 87-04-36 INTERFACE NO: 13

START TIME : 0.0
END TIME : 20.0
DETECTION THRESHOLD : 2.00
MINIMUM PEAK WIDTH : 15.0
AREA REJECT THRESHOLD: 0.0

***** EXTERNAL STANDARD *****

DILUTION FACTOR : 1.0000
SAMPLE VOLUME : 1.0000
STANDARD VOLUME : 1.0000

PK NO	RETEN B TIME C	PEAK NAME	% MEK * 10	ACTUAL G AREA P
1	12.85 2	MEK	10.0103	12364 0

	TOTAL AREA	NO. OF PEAKS
WHOLE SAMPLE:	128683	2
NAMED PEAKS:	12364	1
UN-NAMED PEAKS:	116319	1

***** AREA/HEIGHT PERCENT *****

PK NO	RETEN B TIME C	PEAK AREA	AREA %	PEAK HEIGHT	HEIGHT %
1	12.85 2	12364	9.608	331	10.410
2	14.17 5	116319	90.392	2850	89.590

2 PEAKS; TOTAL AREA: 128683
HEIGHT: 3181

TABLE 3

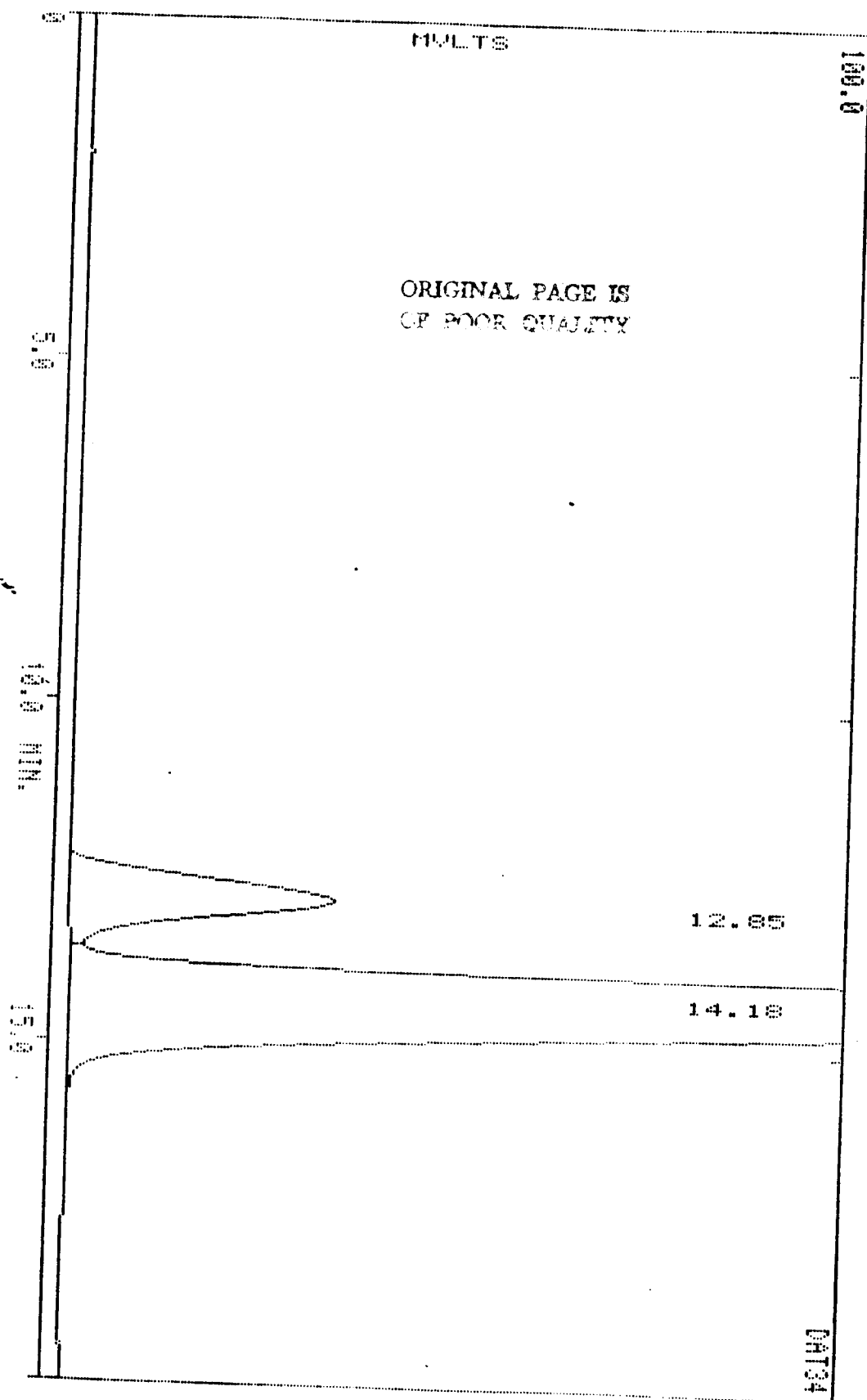


TABLE 1

ORIGINAL PAGE IS
OF POOR QUALITY

***** METHOD TEST4 *****

% PHENOL IN RESINS

DATA ACQUISITION PARAMETERS		
INTERFACE NUMBER		1
DATA CHANNEL		B
DATA START TIME IN MINS.		0.0
DATA END TIME IN MINS.		30.0
PEAK DETECTION THRESHOLD		2.00
MINIMUM PEAK WIDTH IN SECS.		30.0
DISPLAY AND STORE DATA DURING RUN		Y
TIMED EVENTS TABLE REQUIRED		N
MORE DATA PROCESSING PAGES		Y
CALCULATION PARAMETERS		
CALIBRATION BY AREA/HEIGHT		A
SKIM FACTOR		0
CONCENTRATION UNITS	% PHENOL WT/WT	
REPORT GENERATION		
AREA/HEIGHT PERCENT		Y
NORMALISED PERCENT		N
EXTERNAL STANDARD		Y
INTERNAL STANDARD		N
GROUPED REPORT		N
RETENTION TIME WINDOWS		
PERCENT		5
FIXED		15
PEAK NO	RETEN TIME	RESPONSE FACTOR
1	14.00	17.9500
		PEAK NAME
		PHENOL
		GROUP
		0
SAVE DATA		
PLOT FILES		Y
REPORT FILES		Y
PRINT PLOT		Y
DILUTION FACTOR		1.0000
SAMPLE VOLUME		1.0000
STANDARD VOLUME		1.0000
NUMBER OF RUNS		24
CHANNEL B		
SCREEN FULL SCALE MV		100
AREA REJECTION		10
DISC FILE PREFIX		USP27
PRINTED PLOT MIN/PAGE		30
VERTICAL SCALE FACTOR		1

***** METHOD PARAMETERS *****

METHOD: TEST4 : % PHENOL IN RESINS
PLOT: USP01

SAMPLE: USP-39A, #1-1
INJECTION NUMBER: 01

RUN AT: 15:07 ON: 87-03-30 INTERFACE NO: 1B

START TIME : 0.0
END TIME : 19.7
DETECTION THRESHOLD : 2.00
MINIMUM PEAK WIDTH : 30.0
AREA REJECT THRESHOLD: 10.0

***** EXTERNAL STANDARD *****

DILUTION FACTOR : 1.0000
SAMPLE VOLUME : 1.0000
STANDARD VOLUME : 1.0000

PK NO	RETEN TIME	B C	PEAK NAME	% PHENOL	WT/WT	ACTUAL G AREA	G P
3	13.85	1	PHENOL	9.9665		5552	0

	TOTAL AREA	NO. OF PEAKS
WHOLE SAMPLE:	498278	3
NAMED PEAKS:	5552	1
UN-NAMED PEAKS:	492726	2

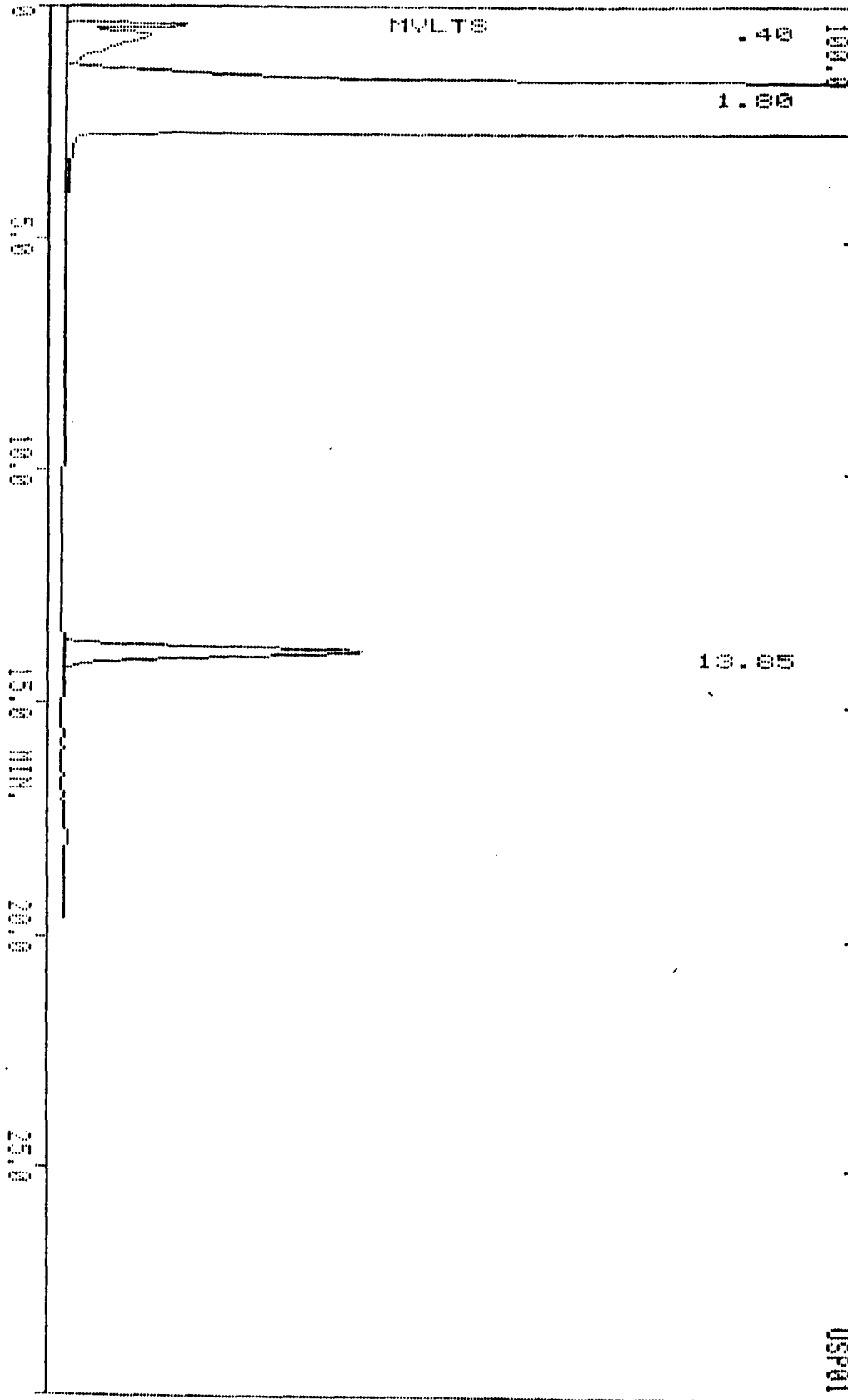
***** AREA/HEIGHT PERCENT *****

PK NO	RETEN TIME	B C	PEAK AREA	AREA %	PEAK HEIGHT	HEIGHT %
1	0.40	2	3606	0.724	189	1.403
2	1.00	5	489119	98.162	12903	95.777
3	13.85	1	5552	1.114	380	2.820

3 PEAKS; TOTAL AREA: 498278
HEIGHT: 13472

TABLE 3

ORIGINAL PAGE IS
OF POOR QUALITY





QUALITY ASSURANCE
TEST METHODS

DATE
3/1/82

NUMBER CTM-57A
Page 1 of 3

SUBJECT: CHEMICAL TEST METHOD FOR
RHEOMETRIC DYNAMIC SPECTROMETRY

PREPARED BY: Fred Bancroft
& James B. Rock

James B. Rock

1.0 SCOPE:

This method describes a procedure for the determination of resin viscosity at various temperatures using the Rheometric Dynamics Spectrometer (RDS) .

2.0 EQUIPMENT AND MATERIALS:

- 2.1 Rheometrics Dynamic Spectrometer with 50 mm parallel plates
- 2.2 Oven, forced air
- 2.3 Balance, 0.1 gm accuracy
- 2.4 Stopwatch
- 2.5 Silicone Release paper
- 2.6 Tongue depressors
- 2.7 RDS systems manual

3.0 SAMPLE:

- 3.1 Raw resins require one (1) oz minimum
- 3.2 Powders require three (3) oz. minimum
- 3.3 Mixed resins require two (2) oz. minimum

4.0 PROCEDURE:

- 4.1 General Test Parameter Capabilities
 - 4.1.1 Temp range-200°C to 395°C linear stepwise heating profile
 - 4.1.2 Strain range is 0 to 100%
 - 4.1.3 Frequency range is 0.01 to 100 radians/sec.
 - 4.1.4 Physical separation of plates (gap) is 0.01 to 5.0 mm
- 4.2 Instrument Set Up (Method I)
 - 4.2.1 Power up instrument and adjust air flow to 45 psi.
 - 4.2.2 Select the following starting front panel parameters:
 - a. Starting temperature - 30°C
 - b. % Strain - 50 (per BMS 8-256)
 - c. Frequency rate - 10 (per BMS 8-256)
 - d. Mode - cure sweep
 - e. Test - Parallel plate
 - 4.2.3 Select operational parameters by typed responses as follows:
 - a. Total time - 140 min.
 - b. Sweep rate - 2.0°C/min.
 - c. 2 min./test point
 - 4.2.4 Press: "Geometry Test"
 - a. Gap - 0.50 mm
 - b. Radius - 25.00 mm



QUALITY ASSURANCE
TEST METHODS

DATE 3/1/82

NUMBER CTM-57A
Page 2 of 3

SUBJECT:
CHEMICAL TEST METHOD FOR
RHEOMETRIC DYNAMIC SPECTROMETRY

PREPARED BY:•
Fred Bancroft
James B. Rock

4.2.5 Press "Plotter"

- a. X-axis min. - 0
- b. X-axis max. - 140
- c. Y-axis min. - 1E0
- d. Y-axis max. - 1E5
- e. η^* - yes, no for all other quantities

4.2.7 Press "Print"

η^*, η', η'' - Torque - yes*
all others - no

*Time and temperature are automatically selected.

4.2.8 Plotter and Paper

1. Insert K & E paper type 46-6210 (5 cy x 7 in)
2. Zero lower left and upper right corners of each chart by adjustment of the zero and venier controls respectively.

4.2.9 Sample Insertion

1. Parallel plates are brought into contact (>10% force normal) and the micrometer set to zero.
2. Sample material should be placed on plates preheated to 70°C and allowed to flow as the gap is set. The plates are then allowed to return to ~30°C.
3. Using the single sweep mode, the resin viscosity is allowed to equilibrate for 2 - 3 minutes at 30°C (% torque is monitored to prevent input overload).

4.2.10 Material Testing Using "Cure Mode" is initiated by the Following Sequence:

1. Temperature = 30°C
2. Mode - Cure
3. Test - Parallel Plate
4. % Strain = 50.00
5. Rate - 10.00 rad/sec
6. START
7. Temperature = 240°C

4.2.11 Viscosity of the resin sample over the temperature range will be plotted and printed. At the desired end of the test, shutdown is as follows:

1. Temperature - 30°C
2. Reset



QUALITY ASSURANCE
TEST METHODS

DATE 3/1/82

NUMBER CTM-57A
Page 3 of 3

SUBJECT:
CHEMICAL TEST METHOD FOR
RHEOMETRIC DYNAMIC SPECTROMETRY

PREPARED BY: •
Fred Bancroft
James B. Rock

4.2.12 Plates may be cleaned by soaking in acetone

5.0 CALCULATION AND REPORTING:

- 5.1 Report RDS viscosity at various temperatures required by the applicable specification.
- 5.2 Attach completed graph with all pertinent information (see para. 4.2.10)



QUALITY ASSURANCE
TEST METHODS

DATE

9/26/86

NUMBER CTM-72

PAGE 1 OF 7

SUBJECT: CHEMICAL TEST METHOD FOR:
PARTICLE SIZE BY THE HORIBA ANALYZER

PREPARED BY: F. Bancroft

APPROVED BY: Hamid M. Quraishi

PARTICLE SIZE

1.0 SCOPE

This procedure describes the method for determining particle size of powders and liquids using the Horiba CAPA-500 Centrifugal Automatic Particle analyzer.

NOTE: The particle size method is in no way an alternative to the manual. The manual should be used in conjunction with the method.

2.0 EQUIPMENT AND MATERIALS

- 2.1 Horiba CAPA-500 Centrifugal Automatic Particle Analyzer
- 2.2 Cuvettes, pyrex with teflon lids, 14 (W) X 14 (D) X 45 (H) mm
- 2.3 Glass Vials, 25-ml with caps
- 2.4 Pipettes, disposable, 10-ml
- 2.5 Pasteur Pipettes
- 2.6 Pipette Bulb
- 2.7 Ultrasound Bath
- 2.8 Beakers, 500-ml
- 2.9 Spatula
- 2.10 Analytical Balance, ± 0.0001 gms
- 2.11 Kimwipes
- 2.12 Ethylene Glycol, 99%+ purity
- 2.13 Glass Microspheres, 1 - 5 μ m
- 2.14 Glass Microspheres, 34.8 μ m
- 2.15 Glass Microspheres, 110.6 μ m
- 2.16 Acetone, industrial
- 2.17 Vacuum

3.0 PREPARATION OF STANDARDS AND SOLUTIONS

3.1 Standards:

3.1.1 Depending on the particle size range desired, there are three glass microspheres standards available:

- a. 1 - 5 μ m
- b. 34.8 μ m
- c. 110.6 μ m

3.1.2 Weigh 10.0 mg of standard into a 25-ml glass vial. Add 20 ml of ethylene glycol. Mix well by shaking, and place vial in ultrasound bath for thirty minutes.



QUALITY ASSURANCE
TEST METHODS

DATE

9/26/86

NUMBER CTM-72

PAGE 2 OF 7

SUBJECT: CHEMICAL TEST METHOD FOR:
PARTICLE SIZE BY THE HORIBA ANALYZER

PREPARED BY: F. Bancroft
APPROVED BY: Hamid M. Quraishi

PARTICLE SIZE, CONTINUED

3.2 Samples:

- 3.2.1 Depending on the samples to be run, each method for sample preparation will be different, namely the solvent used and the concentration of the sample desired; therefore, various addendums have been added at the end of the method to take care of this situation.
- 3.2.2 For carbon black powders and resins, please refer to Addendum 1.
- 3.2.3 For E-03 and E-24 finishes, please refer to Addendum 2.

4.0 PROCEDURE

4.1 Blank:

- 4.1.1 The blank must be run first, before running any standards or samples.
- 4.1.2 Use the same solvent used for the samples to run the blank; e.g. ethylene glycol.
- 4.1.3 Fill both cuvettes with solvent. The liquid level in the cuvette should be between the two engraved lines. Cap the cuvette, and make sure there are no air bubbles.
- 4.1.4 Program the following parameters in the Horiba analyzer:
 - a. Solvent viscosity
 - b. Solvent density
 - c. Sample density
 - d. Diameter (max.)
 - e. Diameter (min.)
 - f. Diameter (div.)
 - g. Centrifugal/Sedimentation Mode
 - h. Speed if centrifugal mode is selected
- 4.1.5 Depending on the samples to be run, each method for particle size analysis will have different values for the parameters described in Section 4.1.3.
- 4.1.6 For carbon black powders and resins, please refer to Addendum 1.
- 4.1.7 For E-03 and E-24 finishes, please refer to Addendum 2.
- 4.1.8 Place one cuvette in reference cell holder, and turn the cell holding bar until the cuvette is fixed.



QUALITY ASSURANCE
TEST METHODS

DATE

9/26/86

NUMBER CTM-72

PAGE 3 OF 7

SUBJECT:

CHEMICAL TEST METHOD FOR:

PARTICLE SIZE BY THE HORIBA ANALYZER

PREPARED BY:

F. Bancroft

APPROVED BY: Hamid M. Quraishi

PARTICLE SIZE, CONTINUED

- 4.1.9 Place the other cuvette in sample cell holder, and turn the cell holding bar until the cuvette is fixed.
- 4.1.10 Close the sample chamber and press the "BLANK" button on the control panel. Measurement is made for thirty-two seconds, and the mean value is stored.
- 4.1.11 Samples are now ready to be run.

4.2 Standards:

- 4.2.1 Fill cuvette with standard, as prepared in Section 3.1. The liquid level in the cell should be between the two engraved lines. Cap the cuvette carefully, making sure no air bubbles are introduced.
- 4.2.2 Place cuvette in sample cell holder, and turn the cell holding bar until the cuvette is fixed.
- 4.2.3 The cuvette in the reference cell holder should contain only ethylene glycol. Secure the reference cell with the cell holding bar.
- 4.2.4 The Horiba analyzer should be programmed in the following manner:
 - a. Solvent viscosity is 19.90 cps
 - b. Solvent density is 1.11 g/cc
 - c. Sample density is 2.30 g/cc
 - d. Diameter (max.) is 10.0 microns
 - e. Diameter (min.) is 0.01 microns
 - f. Diameter (div.) is 1.0 microns
 - g. Speed is 2,000 rpm
 - h. Sample concentration is 0.5 mg/ml
 - i. Running time is 12 minutes, 1 second

The above parameters are for glass microspheres standard, 1 - 5 microns.

- 4.2.5 Close the sample chamber. Press the "SET" button. The running time should appear, in this case, 12 minutes, 1 second.
- 4.2.6 Press the "START" button. Sample is now running. After run stops, data will start printing out.
- 4.2.7 Make sure that the blank is run before running the standard, see Section 4.1.
- 4.2.8 Each standard must be run twice.



QUALITY ASSURANCE
TEST METHODS

DATE

9/26/86

NUMBER CTM-72

PAGE 4 OF 7

SUBJECT:

CHEMICAL TEST METHOD FOR:
PARTICLE SIZE BY THE HORIBA ANALYZER

PREPARED BY: F. Bancroft

APPROVED BY: Hamid M. Quraishi

PARTICLE SIZE, CONTINUED

4.3 Samples:

- 4.3.1 Fill cuvette with sample. The liquid level in the cell should be between the two engraved lines. Cap the cuvette carefully making sure no air bubbles are introduced.
- 4.3.2 Place cuvette in sample cell holder, and secure the cell by turning the cell holding bar.
- 4.3.3 The cuvette in the reference cell holder should contain only solvent, the same solvent used for the sample. Secure the cell by turning the cell holding bar.
- 4.3.4 The Horiba analyzer should be programmed as follows:
 - a. For carbon black powders and resins, refer to Addendum 1.
 - b. For E-03 and E-24 finishes, refer to Addendum 2.
- 4.3.5 Close the sample chamber. Press the "SET" button. The running time should appear.
- 4.3.6 Press the "START" button. Sample is now running. After run stops, data will start printing out.
- 4.3.7 Make sure that the blank is run before running any samples. See Section 4.1.
- 4.3.8 Each sample must be run twice.

5.0 RESULTS

5.1 A typical particle analyzer result is shown in Figure 1.

- 5.1.1 It is crucial that the absorbance, shown in the absorbance vs. time graph, does not exceed 1.0.
- 5.1.2 The average diameter, $D(AVE)$, is based on 50% of the cumulative distribution.
- 5.1.3 The average diameter must not vary by more than 10% from sample-to-sample.
- 5.1.4 The average diameter for the glass microspheres standard, 1 - 5 microns, should be about 4.3 ± 0.1 microns. The value should be entered in the particle size calibration log for future reference.



QUALITY ASSURANCE
TEST METHODS

DATE

9/26/86

NUMBER CTM-72

PAGE 5-OF 7

SUBJECT: CHEMICAL TEST METHOD FOR:
PARTICLE SIZE BY THE HORIBA ANALYZER

PREPARED BY: F. Bancroft

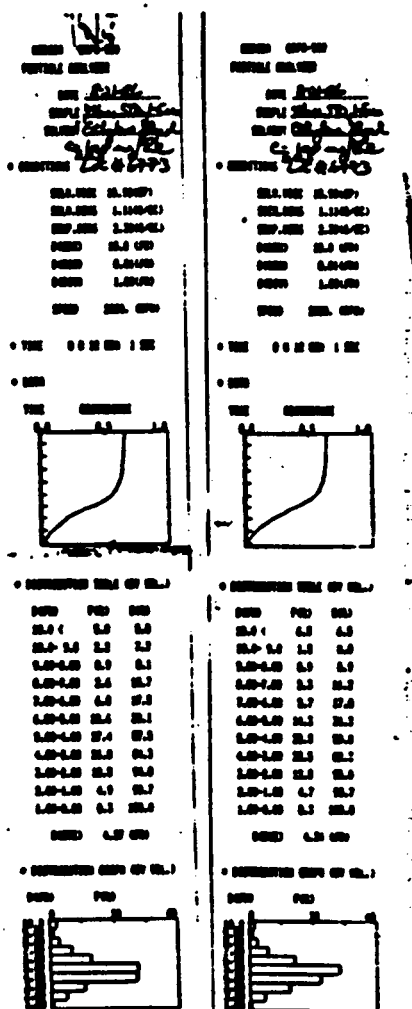
APPROVED BY: Hamid M. Quraishi

PARTICLE SIZE, CONTINUED

ORIGINAL PAGE IS
OF POOR QUALITY

FIGURE 1

PARTICLE SIZE ANALYSIS





QUALITY ASSURANCE
TEST METHODS

DATE

9/26/86

NUMBER CTM-72

PAGE 6 OF 7

SUBJECT: CHEMICAL TEST METHOD FOR:
PARTICLE SIZE BY THE HORIBA ANALYZER

PREPARED BY: F. Bancroft

APPROVED BY: Hamid M. Quraishi

PARTICLE SIZE

ADDENDUM 1

CARBON BLACK POWDERS AND RESINS SAMPLE PREPARATION

1.0 SAMPLE PREPARATION

- 1.1 Weigh 2 mg of sample in a 25-ml vial. Add 20 ml of ethylene glycol; concentration is 0.1 mg/ml. Mix well in ultrasound bath for thirty minutes.
- 1.2 Take 2 ml of 0.1 mg/ml solution and add 20 ml of ethylene glycol in a 25-ml vial; concentration is 0.01 mg/ml. Mix well in ultrasound bath for thirty minutes.

2.0 HORIBA ANALYZER DATA

The following data is needed for running the carbon black samples:

- 2.1 Solvent is ethylene glycol
- 2.2 Solvent viscosity is 19.90 cps
- 2.3 Solvent density is 1.11 g/cc
- 2.4 Sample density is 1.90 g/cc
- 2.5 Diameter (max.) is 5.0 microns
- 2.6 Diameter (min.) is 0.01 microns
- 2.7 Diameter (div.) is 0.50 microns
- 2.8 Speed is 5,000 rpm
- 2.9 Sample concentration is 0.01 mg/ml
- 2.10 Running time is 11 minutes, 31 seconds



QUALITY ASSURANCE
TEST METHODS

DATE

9/26/86

NUMBER CTM-72

PAGE 7 OF 7

SUBJECT: CHEMICAL TEST METHOD FOR:
PARTICLE SIZE BY THE HORIBA ANALYZER

PREPARED BY: F. Bancroft

APPROVED BY: Hamid M. Quraishi

PARTICLE SIZE

ADDENDUM 2

E-03 AND E-24 FINISHES

1.0 SAMPLE PREPARATION

- 1.1 Weigh 120 mg of sample in a 25-ml vial if the percent solids is 20%. Add 20 ml of deionized water; concentration is 6.0 mg/ml. Mix well in ultrasound bath for thirty minutes.
- 1.2 Weigh 60 mg of sample in a 25-ml vial if the percent solids is 40%. Add 20 ml of deionized water; concentration is 3.0 mg/ml. Mix well in ultrasound batch for thirty minutes.

2.0 HORIBA ANALYZER DATA

The following data is needed for running the E-03 and E-24 finishes:

- 2.1 Solvent is deionized water
- 2.2 Solvent viscosity is 0.94 cps
- 2.3 Solvent density is 0.99 g/cc
- 2.4 Sample density is 1.25 g/cc
- 2.5 Diameter (max.) is 2.0 microns
- 2.6 Diameter (min.) is 0.01 microns
- 2.7 Diameter (div.) is 0.25 microns
- 2.8 Speed is 5,000 rpm
- 2.9 Sample concentration is 6.0 mg/ml (20% solids), or 3.0 mg/ml (40% solids)
- 2.10 Running time is 6 minutes, 34 seconds

TABLE OF CONTENTS
PHYSICAL TEST METHODS
NAS8-36298
U.S. POLYMERIC O.E. 71108

<u>Number</u>	<u>Subject</u>
PTM-3A	Fabric Weight
PTM-5A	Fabric Thread Count
PTM-7C	Solids of Resin Solutions
PTM-14C	Brookfield Viscosity
PTM-16F	Wet and Dry Resin Contents using Carrier Weight
PTM-17B	Volatile Content of Prepreg Materials
PTM-19G	Wet and Dry Flow by Flash Removal or Punched Disk
PTM-20E	Gel Time of Prepreg Materials
PTM-29C	Specific Gravity of Liquids, Semi-Solids and Solids by Pycnometer
PTM-29C	Amplifying Remarks
PTM-47B	Gel Time by Oil Bath
PTM-61B	Linear Thermal Expansion
PTM-71B	Ash Content of Fillers and Related Materials
PTM-80	Minimum Tack Temperature for Pattern Grade Prepreg
PTM-84	Specific Gravity of Solids by "Air Comparison" Pycnometer
PTM-84	Amplifying Remarks
PTM-98	Determination of Press Laminate Residual Volatiles

NOTE: Individual pages are not numbered except in sequence
for each test method.

SUBJECT:

PHYSICAL TEST METHOD FOR:
FABRIC WEIGHT

APPROVED

James B. Rock

*Sam**JBR**HMT*

1.0 Scope

- 1.1 This method describes a procedure for determination of fabric weight using small specimens. Unconditioned, conditioned, and devolatilized methods are included.
- 1.2 The procedure is equivalent to CCC-T-191, Method 5041.

2.0 Equipment

- 2.1 Scissors
- 2.2 Analytical balance
- 2.3 Steel template, 6.76 inches square
- 2.4 Stanley knife or equivalent
- 2.5 Desiccator with conditioning chemicals giving a relative humidity of $65 \pm 2\%$
- 2.6 Oven capable of maintaining $70^\circ \pm 3^\circ\text{F}$
- 2.7 Desiccator with silica gel or equivalent
- 2.8 Timer
- 2.9 Oven, forced air

3.0 Sample Size

- 3.1 One linear yard shall be taken for test.


4.0 Procedure

- 4.1 From the sample, cut at random five (5) specimens each 6.76 inches square. No specimen shall be closer than three inches to the selvage. Weigh individual specimens to 0.01 gm.
- 4.2 If the conditioned method is used, precondition specimens for 24 ± 2 hours at $65 \pm 2\%$ relative humidity and $70^\circ \pm 3^\circ\text{F}$. Weigh individual specimens to 0.01 gm.
- 4.3 If devolatilized method is used, omit paragraph 4.2 and suspend specimens in a forced air oven at $320^\circ \pm 5^\circ\text{F}$ for 15 ± 0.25 minutes. Weigh individual specimens to 0.01 gm.

5.0 Calculations and Reporting

- 5.1 The individual specimen weight in grams is equivalent to its weight in ounces per square yard.
- 5.2 To calculate unconditioned fabric weight (as received), obtain the average of the five values of paragraph 4.1. Designate as Unconditioned Fabric Weight, W_1 . The range of the five fabric weights should not exceed 3.0%.
- 5.3 To calculate conditioned fabric weight, obtain the average of the five values of paragraph 4.2. Designate as Conditioned Fabric Weight, W_2 . The range of the five fabric weights should not exceed 2.0%.
- 5.4 To calculate devolatilized fabric weight, obtain the average of the five values of paragraph 4.3. Designate as Devolatilized Fabric Weight, W_3 . The range of the five fabric weights should not exceed 2.0%.
- 5.5 To calculate volatile content, use the following formula:

$$\% \text{ Volatile Content} = \frac{W_1 - W_3}{W_1} \times 100$$

 P.O.S. Chemicals, Inc.		QUALITY ASSURANCE TEST METHODS	DATE 6/13/66	NUMBER PTM-5A Page 1 of 1
SUBJECT: <div style="border: 1px solid black; display: inline-block; padding: 2px; margin-top: 5px;">A</div> PHYSICAL TEST METHOD FOR: FABRIC THREAD COUNT		APPROVED <div style="text-align: right; margin-top: 10px;"> <i>P.D. McCart</i> 6/13/66 PREPARED BY: * P. D. McCart </div>		
APPROVAL SIGNATURES:				
GENERAL MANAGER DATE N/A	ENGINEERING DATE N/A	PRODUCTION DATE N/A	PRODUCTION CONTROL DATE N/A	
PURCHASING DATE N/A	SALES DATE N/A	QUALITY ASSURANCE* <i>H. m. Toellner</i> DATE 13 June 1966		

1. SCOPE
 - 1.1 Scope. This method describes a procedure for the determination of the number of warp and fill yarns per inch of woven cloth.
 - 1.2 Equivalent method. This method is similar to CCC-T-191b, Method 5050.1.
2. EQUIPMENT AND MATERIALS
 - 2.1 Equipment.
 (a) Thread counter apparatus with magnifier
3. SAMPLING
 - 3.1 Sample size. Sufficient sample shall be obtained so as to permit specified amount of determinations to be made in each the warp and fill directions.
 - 3.2 Determinations. Unless otherwise specified, five determinations will be made in each the warp and fill directions.
4. PROCEDURE
 - 4.1 Preparation of sample. The cloth shall be laid out smoothly and without tension on a horizontal surface.
 - 4.2 Determination of thread count.
 - 4.2.1 Area of determination. The actual count shall be determined from representative sections of the cloth along the warp and fill directions. No count shall be made nearer than 3 inches to the selvage edge, or either end, and no two determinations should include the same threads.
 - 4.2.2 Inches for each count. If there are more than 25 threads per inch, count one inch for each determination; if there are fewer than 25 threads per inch, count three inches per each determination made.
5. CALCULATIONS AND REPORTING
 - 5.1 Average the specified number of determinations for each warp and fill direction and report these values, rounded off to the nearest whole number.

*Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY ASSURANCE
TEST METHODS

DATE

4/17/67

NUMBER PTM-7C

Page 1 of 3

SUBJECT:



PHYSICAL TEST METHOD FOR:
SOLIDS OF RESIN SOLUTIONS

PREPARED BY: *WCK*

76-07

P. H. Davis

P. H. Davis 4/20/67

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE

N/A

DATE

N/A

DATE

N/A

DATE

N/A

PURCHASING

N/A

SALES

N/A

DATE

DATE

QUALITY ASSURANCE*

P. H. Davis 4-25-67
DATE 4-25-67

1. SCOPE

- 1.1 This method describes a procedure for the determination of total resin solids content of liquid solutions.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Balance, analytical, minimum sensitivity 0.001 gms.
- (b) Desiccator
- (c) Hypodermic syringe, 2 ml. or 5 ml.
- (d) Oven, forced air (see 6.1)

2.2 Materials.

- (a) Aluminum weighing dish, diameter 2-3/8 inch, depth 5/8 inch, weight approximately 1.4 gms.
- (b) Eyedroppers

3. SAMPLING

- 3.1 Sample size. Unless otherwise specified, use one gram of resin solids for each determination. To find the exact sample size, the formula $1/(\text{total solids})(1-\text{filler content})$ will yield the correct weight to use. The following table shall serve as a guide when the anticipated solids are known(unfilled only):

ANTICIPATED SOLIDS

SAMPLE SIZE, GMS.

20%	5.00 + 0.5
30	3.30 + 0.3
40	2.50 + 0.2
50	2.00 + 0.2
60	1.70 + 0.2
70	1.45 + 0.1
80	1.25 + 0.1
90	1.10 + 0.1
100	1.00 + 0.1

When the anticipated solids are not known, run one set of solids at the 70% concentration. Once the solids are determined, conduct an additional test using the correct sample size.

* Signatures necessary for all Quality Assurance Procedures and Instructions.

QUALITY ASSURANCE
TEST METHODS

DATE

4/17/67

NUMBER

PTM-7C

Page 2 of 3

SUBJECT:

PHYSICAL TEST METHOD FOR:
SOLIDS OF RESIN SOLUTIONSPREPARED BY: *W. H. Davis*P. H. Davis *P. H. Davis 4/20/67*

3.2 Number of determinations. Unless otherwise specified, determine total resin solids in triplicate.

4. PROCEDURE

4.1 Weighing. Weigh an aluminum dish to nearest 0.001 gram. The following procedure shall be used for transferring and weighing the resin.

4.1.1 Low viscosity resins. For low viscosity resins, especially resins using acetone as a solvent, use a syringe. Rapidly fill the syringe and wipe off the outside tip. Weigh syringe and resin to nearest 0.001 gm. Expel the approximate weight of sample (see 3.1) from the syringe into the aluminum dish. Retract the syringe piston and reweigh the syringe to nearest 0.001 gm.

4.1.2 High viscosity resins. For high viscosity resins, use an eyedropper. Quickly discharge the sample into the aluminum dish. Immediately weigh the resin and aluminum dish to nearest 0.001 gm.

4.2 Spreading sample. Tilt and rotate the aluminum dish to spread resin over entire bottom of dish. Warm the resin on a hot plate if necessary to accomplish spreading (see note 6.2). Add approximately 6 ml. of toluene to SMR dispersions.

4.3 Devolatilization. Unless otherwise specified, place samples in an air circulating oven for time and temperature indicated below (see note 6.3):

RESIN TYPE	TEMPERATURE, °F	TIME, MINUTES
Epoxy	320 ± 5	45 ± 2
Polyester	275 ± 5	20 ± 1
Phenolic	275 ± 5	60 ± 2
Phenyl-Silane	275 ± 5	60 ± 2
Silicones	275 ± 5	180 ± 3
SMR Dispersions	275 ± 5	60 ± 2

4.4 Reweighing. Remove dishes from oven and cool in a desiccator (see note 6.4). Reweigh each aluminum dish and residue to nearest 0.001 gm.




5. CALCULATIONS AND REPORTING

5.1 Calculations. Calculate solids as follows:

5.1.1 Syringe method.

$$\text{Solids, wt. \%} = \frac{W_2 - W_1}{W_4 - W_3} \times 100$$

where: W_1 = weight of aluminum dish, gms.
 W_2 = weight of dish and residue, gms.
 W_3 = weight of syringe, gms., after expelling sample portion
 W_4 = weight of syringe and sample, gms.

	QUALITY ASSURANCE TEST METHODS	DATE 4/17/67	NUMBER PTM-7C Page 3 of 3
SUBJECT:  PHYSICAL TEST METHOD FOR: SOLIDS OF RESIN SOLUTIONS		PREPARED BY: *  P. H. Davis <i>P.H. Davis</i> 4/24/67	

5.1.2 Eyedropper method.

$$\text{Solids, wt. \%} = \frac{W_2 - W_1}{W_5 - W_1} \times 100$$

where: W_5 = weight of dish and resin sample, gms.

5.2 Reporting. Unless otherwise specified, round-off and report the average of three determinations to 0.1%. Triplicate determinations should be considered suspect if the range from the average exceeds 0.3% solids.

6. NOTES

- 6.1 Oven requirements. The oven should have a wattage range of between 1200 and 4000 watts, and an air current between 600 and 1500 feet per minute when measured at the internal ports with an air flow meter (velometer).
- 6.2 Spreading sample. The sample should remain in the hot plate only as long as is necessary to make the resin liquid enough to spread. A low temperature hot plate should be used, preferably lower than 275°F.
- 6.3 Placing samples in oven. Aluminum dishes should be placed in the center of the oven with no more than nine (9) samples placed in the oven at one time. The time required for opening the door, inserting aluminum dish(es) and closing the door should not exceed ten (10) seconds.
- 6.4 Precautions in removing samples from oven. In removing samples which have bubbled from the oven, precautions should be taken to prevent loss of sample from spattering.

ORIGINAL PAGE IS
OF POOR QUALITY



QUALITY ASSURANCE
TEST METHODS

DATE
5/12/82

NUMBER PTM-14C

Page 1 of 4

SUBJECT:

PHYSICAL TEST METHOD FOR:
BROOKFIELD VISCOSITY

PREPARED BY:*

James B. Rock
James B. Rock

1. SCOPE

- 1.1 This is a method for the determination of viscosity of liquids using a Brookfield Viscometer. (Method I)
- 1.2 Also included is the Brookfield Thermosel System which is designed for measuring temperatures over a range of 100°F to 500°F. (Method II)

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- (a) Brookfield Viscometer, Model RVF with 7 spindles (for both methods)
- (b) Thermometer, range 74°F to 79°F in 0.1°F divisions (for Method I)
- (c) Water bath, capable of maintaining 77° ± 0.5°F (for Method I)
- (d) Thermo-container and controller (for Method II)

3. SAMPLING

- 3.1 Materials having ingredients which tend to settle should be thoroughly mixed before sampling. A representative one-quart sample is required for each determination.

4. PROCEDURE

4.1 Method I

- 4.1.1 Sample conditioning Place approximately one quart of sample in a round container with a sufficient diameter and depth so that the spindle will not be closer than one inch at any point to the container. Place the sample in a water bath and condition to 77° ± 0.5°F, unless otherwise specified.

4.1.2 Determination of viscosity

- (a) Level the viscometer by means of tripod screws and bubble level.
- (b) Select a spindle consistent with expected viscosity and screw it into the viscometer using counter-clockwise rotation.
- (c) Immerse the spindle into the liquid to the depth where the notch on the stem is even with the liquid level.
- (d) Unless otherwise specified, rotate the spindle at 20 RPM, depressing the clutch. Release the clutch after one revolution and read the outer dial as soon as equilibrium is established.
- (e) If dial reading is less than 10, repeat test using a smaller numbered spindle; if greater than 90, repeat using a larger numbered spindle. Make two readings.
- (f) Turn viscometer off, remove sample and clean spindle thoroughly.



QUALITY ASSURANCE
TEST METHODS

DATE
5/12/82

NUMBER PTM-14C
Page 2 of 4

SUBJECT:

PHYSICAL TEST METHOD FOR:
BROOKFIELD VISCOSITY

PREPARED BY:•

James B. Rock

4.2 Method II

4.2.1 Set Up Viscometer

Set up the viscometer stand connecting the black 3-1/4 inch coupling post to the rack. Screw both posts into the base leaving the lock nut loose. Put the three (3) leveling screws into position on the base. Attach the viscometer to the stand inserting it into the clamp, and position it reasonably level and centered between the stand legs. Lock the posts tight to the stand base with the lock nut on the coupling post. Raise the viscometer to the highest position on the stand. Check the power switch off and plug the viscometer power cord into a 15 amp, 115 volt, 60 cycle A.C. electrical service.

4.2.2 Set Up Controller

Set controller on a level surface adjacent to thermo-container. Insert the male plug (three-prong) from the thermo-container braided cord into the socket on the back of the controller (turn and lock connection). Insert the four inch stainless steel probe (Resistance Thermometer) into the hole in the thermo-container located directly above the braided cord. Plug the other end of the probe into the connector located on the back of the controller. To remove probe, depress spring clip and carefully slide probe out. The procedure should not be followed if the thermo-container is at a temperature above 100°F. NOTE: If the probe is NOT inserted in the thermo-container and the controller is turned on, the thermocontainer will be destroyed.

4.2.3 Familiarization

The operator should become familiar with the alignment procedure so that he can safely align and operate the system at elevated temperatures. The system is designed to operate in the temperature range from ambient to a maximum of 500°F (260°C). Precise control of test sample temperature is possible in the 100°F to 500°F range with accuracy of $\pm 0.5\%$ of the controller set point. That is, at 500°F set point, the test sample temperature will be held to $\pm 2.5^\circ\text{F}$.

4.2.4 Determination of Viscosity

- (a) Remove the insulating cap and spindle.
- (b) Raise the viscometer to the highest level on the stand.
- (c) Remove the sample chamber, using the extracting tool, and place it in the auxiliary holder.
- (d) Using a syringe, graduate or other suitable measuring device, pour into the sample chamber the volume of liquid sample designated on the data sheet. DO NOT OVERFILL! The liquid level should intersect the spindle shaft at a point approximately 1/8 inch above the upper "Conical Body" - "Spindle Shaft" Interface.



QUALITY ASSURANCE
TEST METHODS

DATE

5/12/82

NUMBER PTM-14C

Page 3 of 4

SUBJECT:

PHYSICAL TEST METHOD FOR:
BROOKFIELD VISCOSITY

PREPARED BY: •

James B. Rock

- (e) Using the extracting tool, put the loaded chamber back into the thermo-container.
- (f) Lower the viscometer and align the thermo-container.
- (g) Insert the spindle into the liquid in the chamber and couple it to the viscometer (observe left hand thread).
- (h) Replace the insulating cap.
- (i) Set the control knob on the controller to the desired set point temperature at which viscosity measurements are to be made. The Controller scale reads directly in the 0-500°F range with 1°F increments.
- (j) For temperatures between 100°F and 250°F, set the control knob initially 20° LOWER than the desired equilibrium temperature (for temperatures between 250°F and 500°F, set control knob on temperature desired). This will prevent temperature overshoot in the thermo-container and reduce the lag time to reach spindle, chamber and sample temperature equilibrium. As the temperature rises in the thermo-container, the power output level light will go from full ON to dimmer and then full OFF. When the temperature in the thermo-container has dropped to the initial set point (after about 5 minutes), the output level light will start to glow. Advance the set point knob in consecutive steps up to the desired setting. About 30 minutes should be allowed for system temperature equilibrium.
EQUILIBRIUM CONDITIONS WILL BE DENOTED BY A STEADY GLOW OF THE OUTPUT LEVEL LIGHT ON THE CONTROLLER, STABILIZED DEVIATION METER AND BY A STEADY DIAL READING ON THE VISCOMETER.
- (k) Turn ON the Viscometer and leave it running during the equilibrium period. The rotating spindle provides agitation which reduces temperature equilibrium time and temperature gradients within the test sample.
- (l) Turn on the controller and refer to 4.2.4 (i) above prior to taking viscosity measurements.
- (m) After the thermo-container, spindle, chamber and test sample has reached temperature equilibrium, viscometer readings are taken at different speeds and viscosities are obtained using the ranges supplied on the data sheet.

5. CALCULATIONS AND REPORTING

5.1 Calculations for Method I

Calculate viscosity as follows:

$$\text{Viscosity in centipoise} = RF$$

where: R = average dial reading

F = factor from the following chart:



L. S. Polymeric, Inc.

QUALITY ASSURANCE
TEST METHODSDATE
5/12/82

NUMBER PTM-14C

Page 4 of 4

SUBJECT:

PHYSICAL TEST METHOD FOR:
BROOKFIELD VISCOSITY

PREPARED BY: *

James B. Rock

<u>Spindle No.</u>	<u>20 RPM Factor</u>	<u>10 RPM Factor</u>	<u>4 RPM Factor</u>	<u>2 RPM Factor</u>
1	5	10	25	50
2	20	40	100	200
3	50	100	250	500
4	100	200	500	1,000
5	200	400	1,000	2,000
6	500	1,000	2,500	5,000
7	2,000	4,000	10,000	20,000

5.2 Calculations for Method II

<u>Speed</u> <u>(RPM)</u>	<u>Spindle Number</u>			
	21	27	28	29
100	5	25	50	100
50	10	50	100	200
20	25	125	250	500
10	50	250	500	1M
5	100	500	1M	2M
4	125	625	1.25M	2.5M
2.5	200	1M	2M	4M
2	250	1.25M	2.5M	5M
1	500	2.5M	5M	10M
0.5	1M	5M	10M	20M

5.3 Reporting for Both Methods I and II

Report viscosity in centipoise or poise (as applicable) to three significant figures. The test results should be considered suspect if the range for duplicate readings exceeds 0.2%.



QUALITY ASSURANCE
TEST METHODS

DATE

8/10/66

NUMBER PTM-16F

Page 1 of 3

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY RESIN CONTENTS
USING CARRIER WEIGHT

APPROVED

P.D.

PREPARED BY:*

P. D. McCart 8/22/66

APPROVAL SIGNATURES:

GENERAL MANAGER

N/A

DATE

ENGINEERING

N/A

DATE

PRODUCTION

N/A

DATE

PRODUCTION CONTROL

N/A

DATE

PURCHASING

N/A

DATE

SALES

N/A

DATE

QUALITY ASSURANCE*

H. M. Toellner
DATE *23 Aug 1966*

1. SCOPE

1.1 Scope. This method describes the procedure and equipment necessary to determine the wet and dry resin content of all fabric, paper, and roving prepreps using carrier weights.

1.2 Classification. The resin content calculations shall be of the following types:

- Type I - Dry resin content based on dry carrier weight
- Type II - Dry resin content based on carrier weight
- Type III - Wet resin content based on dry carrier weight
- Type IV - Wet resin content based on carrier weight
- Type V - Wet resin solids based on dry carrier weight
- Type VI - Wet resin solids based on carrier weight

2. EQUIPMENT

(F) 2.1 Equipment.

- (a) Laboratory knife
- (b) Steel template
- (c) Analytical balance
- (d) Hooks, "S" shaped
- (e) Oven, forced air

3. SAMPLING

3.1 Sample location. The carrier sample shall be taken from each roll of material used in the impregnation. The carrier sample should be taken from an area adjacent to the prepreg sample.

3.2 Sample size. The sample size applies to the carrier and the impregnated material. The sample of fabrics or paper should be approximately 8 inches by the width of the material and for roving should be approximately 30 linear feet.

(F) 3.3 Number of determinations. Unless otherwise specified, conduct three determinations on each sample.

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY RESIN CONTENTS
USING CARRIER WEIGHT

APPROVED

PREPARED BY: P. D. McCart 8/22/66

4. PROCEDURE

- F** 4.1 Specimen preparation. Using the steel template and laboratory knife, cut three representative specimens from the sample, unless otherwise specified.
(a) Roving specimens should be 72 ± 0.1 inches long.
(b) Fabric and paper specimens should be approximately 4 inches square.
- 4.2 Carrier weight. The carrier and dry carrier weight shall be determined as follows:
- 4.2.1 Carrier weight. Weigh each unimpregnated specimen and record this weight as the carrier weight. (W_0)
- 4.2.2 Dry carrier weight. Determine the carrier weight, then devolatilize the specimens in an air circulating oven at $325 \pm 5^\circ\text{F}$ for 10 minutes unless otherwise specified. Cool the specimens in a desiccator, then weigh. Record the devol weight of each specimen as the dry carrier weight. (W_1).
- 4.3 Prepreg weight. The prepreg weight and dry prepreg weight shall be determined as follows:
- 4.3.1 Prepreg weight. Weigh each impregnated specimen and record this weight as the prepreg weight. (W_2)
- 4.3.2 Dry prepreg weight. Determine the prepreg weight, then devolatilize the specimens per PTM-17, the customer specification, or the applicable procedures. Cool the specimens in a desiccator, then weigh. Record the devol weight of each specimen as the dry prepreg weight. (W_3)

5. CALCULATIONS AND REPORTING

- 5.1 Calculations. The calculations for resin content shall be as follows:

- 5.1.1 Type I, Dry Resin Content. Type I, Dry Resin Content shall be determined as follows:

$$\text{Percent Dry Resin Content (DRC)} = \frac{(W_3 - W_1)F \times 100}{W_3}$$

(Dry carrier basis)

where: W_1 = dry carrier weight, gms.
 W_3 = dry prepreg weight, gms.
 F = factor for prepreg containing filler

- 5.1.2 Type II, Dry Resin Content. Type II, Dry Resin Content shall be determined as follows:

$$\text{Percent Dry Resin Content (DRC)} = \frac{(W_3 - W_0)F \times 100}{W_3}$$

("As is" carrier basis)

where: W_0 = carrier weight, gms.
 W_3 = dry prepreg weight, gms.
 F = factor for prepreg containing filler

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY RESIN CONTENTS
USING CARRIER WEIGHT

APPROVED

PREPARED BY: P. D. McCart 8/22/66

5.1.3 Type III, Wet Resin Content. Type III, Wet Resin Content shall be determined as follows:

$$\text{Percent Wet Resin Content (WRC)} = \frac{(W_2 - W_1)F \times 100}{W_2}$$

(Pickup - dry carrier basis)

where: W_1 = dry carrier weight, gms.
 W_2 = prepreg weight, gms.
 F = factor for prepreg containing filler

5.1.4 Type IV, Wet Resin Content. Type IV, Wet Resin Content shall be determined as follows:

$$\text{Percent Wet Resin Content (WRC)} = \frac{(W_2 - W_0)F \times 100}{W_2}$$

(Pickup - "As is" carrier basis)

where: W_0 = carrier weight, gms.
 W_2 = prepreg weight, gms.
 F = factor for prepreg containing filler

5.1.5 Type V, Wet Resin Solids. Type V, Wet Resin Solids shall be determined as follows:

$$\text{Percent Wet Resin Solids (WRS)} = \frac{(W_3 - W_1)F \times 100}{W_2}$$

(Dry carrier basis)

where: W_1 = dry carrier weight, gms.
 W_2 = prepreg weight, gms.
 W_3 = dry prepreg weight, gms.
 F = factor for prepreg containing filler

5.1.6 Type VI, Wet Resin Solids. Type VI, Wet Resin Solids shall be determined as follows:

$$\text{Percent Wet Resin Solids (WRS)} = \frac{(W_3 - W_0)F \times 100}{W_2}$$

("As is" carrier basis)

where: W_0 = carrier weight, gms.
 W_2 = prepreg weight, gms.
 W_3 = dry prepreg weight, gms.
 F = factor for prepreg containing filler

F 5.2 Reporting. Unless otherwise specified, report resin content to 0.1%.

6. NOTES

6.1 Definitions. The following terms are defined for clarity.

- 6.1.1 Dry Resin Content. Dry Resin Content is the same as dry resin solids, commonly abbreviated as DRC.
- 6.1.2 Wet Resin Content. Wet Resin Content is the same as resin pickup, commonly abbreviated as WRC.
- 6.1.3 Wet Resin Solids. Term assigned to the intermediate determination as in 5.1.5 and 5.1.6, abbreviated as WRS.



QUALITY ASSURANCE
TEST METHODS

DATE

3/21/78

NUMBER

PTM-17B

Page 1 of 3

SUBJECT:

PHYSICAL TEST METHOD FOR:
VOLATILE CONTENT OF PREPREG MATERIALS

PREPARED BY:*

James B. Rock

James B. Rock

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING

SALES

QUALITY ASSURANCE*

DATE N/A

DATE N/A

DATE

1. SCOPE

1.1 The purpose of this method is to describe a procedure for the determination of volatile content of preimpregnated materials.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- (a) Steel templates, 4 inches by 4 inches and 2 inches by 2 inches
- (b) Hooks, "S" shaped
- (c) Oven, forced air
- (d) Oven, dead air
- (e) Mettler balance, or equivalent, capable of weighing to 0.001 gram
- (f) Stanley knife or equivalent
- (g) Wide mouth ceramic crucibles, Coors No. 1 or equivalent
- (h) Desiccator

3. SAMPLING

3.1 Sample size

3.1.1 Prepregs other than unidirectional tapes. Three specimens 4 inches by 4 inches are cut randomly from a representative sample one foot by width of prepreg.

3.1.2 Unidirectional tapes. Three specimens 2 inches by 2 inches (or equivalent of 4 square inches per specimen) shall be randomly cut from a sample of representative of the material.

4. PROCEDURE

4.1 Prepregs other than unidirectional tapes.

4.1.1 Preparing samples. Cut three 4 inch by 4 inch specimens using steel template and weigh collectively to the nearest 0.001 gm.

4.1.2 Testing samples. Attach "S" shaped hook to one corner of each specimen and place the specimens in the designated type of oven for required time and temperature as per the chart in Para. 4.2.2. Suspend the specimens individually, allowing room for free air circulation between each. Do not allow the door to remain open longer than 10 seconds while loading the specimens. If dripping of

* Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY ASSURANCE
TEST METHODS

DATE

3/21/78

NUMBER PTM-17B

Page 2 of 3

SUBJECT:
PHYSICAL TEST METHOD FOR:
VOLATILE CONTENT OF PREPREG MATERIALS

PREPARED BY: •

James B. Rock

4.1.2 (Cont'd)

resin occurs repeat, hanging each specimen by diagonal corners. Remove the specimens from the oven, desiccate for 5 minutes and reweigh collectively to the nearest 0.001 gm. Desiccation may be waived unless specifically required by customer specification or test method, provided the weighing is done immediately upon removal of the specimens from the oven.

4.2 Unidirectional tapes.

4.2.1 Preparing samples. Cut three 2 inch by 2 inch (or equivalent) specimens and weigh each into a previously tared crucible, to the nearest 0.001 gm.

4.2.2 Testing samples. Place the crucibles in the designated type of oven for the required time and temperature as per the chart. Do not allow the oven door to remain open longer than 10 seconds while loading oven.

<u>PREPREG TYPE</u>	<u>* TEMPERATURE, °F</u>	<u>** TIME, MINUTES</u>
Phenolics or Phenylsilanes	320	9
Polyesters	220	8, Dead Air Oven
Standard Epoxies	320	9
Special Epoxies	275	15
Silicones	320	5
Paper, Melamines	320	8, Dead Air Oven
Paper, Polyesters	220	10, Dead Air Oven
Polyimides	450	10
Miscellaneous	**	**

* All temperature tolerances are $\pm 5^{\circ}\text{F}$; all time tolerances are ± 0.25 minute. All ovens are forced air except as noted.

** For miscellaneous prepreg types, temperature and time are as per customer specification.

Remove the crucibles from the oven, desiccate for 5 minutes and reweigh each to the nearest 0.001 gm.

5. CALCULATIONS AND REPORTING

5.1 Prepregs other than unidirectional tape.



QUALITY ASSURANCE
TEST METHODS

DATE

3/21/78

NUMBER PTM-17B

Page 3 of 3

SUBJECT:

PHYSICAL TEST METHOD FOR:
VOLATILE CONTENT OF PREPREG MATERIALS

PREPARED BY: •

James B. Rock

5.1.1 Calculate volatile content as follows:

$$\% \text{ Volatile} = \frac{W_1 - W_2}{W_1} \times 100$$

Where: W_1 = Original weight, gms.
 W_2 = Final weight, gms.

5.2 Unidirectional tapes.

5.2.1 Calculate volatile content as follows:

$$\% \text{ Volatile} = \frac{(W_1 - W_c) - (W_2 - W_c)}{W_1 - W_c} \times 100$$

Where: W_1 = Weight of crucible and original sample

W_c = Weight of crucible

W_2 = Weight of crucible and sample from oven

5.3 Repeated tests should agree within 0.2%.



QUALITY ASSURANCE
TEST METHODS

DATE Revised
April 28, 1975

NUMBER PTM-19-G
Page 1 of 6

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY FLOW BY FLASH
REMOVAL OR PUNCHED DISC

PREPARED BY: * *J. W. Mansfield*
J. W. Mansfield

APPROVAL SIGNATURES:

GENERAL MANAGER	ENGINEERING	PRODUCTION	PRODUCTION CONTROL
DATE N/A	DATE N/A	DATE N/A	DATE N/A
PURCHASING	SALES		QUALITY ASSURANCE*
DATE N/A	DATE N/A		<i>J. W. Mansfield</i> DATE 4-29-75

1. SCOPE

- 1.1 This is a method for the determination of flow by either flash removal or punched disc. Both wet (prepreg tested as is) and dry (devolatilized) flows are incorporated.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Laboratory press, hydraulic
- (b) Analytical balance
- (c) Caul plates, stainless steel, 1/16" thick, 6" x 6" (optional)
- (d) Steel templates, 2" x 2" and 4" x 4"
- (e) Laboratory knife
- (f) Punch press with cutting die, 2,500 diameter
- (g) Calipers, graduated in 0.01 inch

2.2 Materials.

- (a) Release films, cellophane, mylar, aluminum foil, teflon, coated fabric.
- (b) TX-1040 light weight teflon coated glass fabric, or equivalent
- (c) Mochburg Fabric, Grade CW-1850 (West Coast Paper Co., Seattle, Wash.) or equivalent.

3. SAMPLING

- 3.1 Sample size. The sample shall be large enough so that the required number of 4" x 4" plies may be cut. Unless otherwise specified, cut specimens on the bias. The total specimen weight should be 25 to 30 grams, except in the case of the high modulus graphite tape. The following table will serve as a guide in determining the number of plies per specimen:

* Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY ASSURANCE
TEST METHODS

DATE Revised
April 28, 1975

NUMBER PTM-19-G
Page 2 of 6

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY FLOW BY FLASH
REMOVAL OR PUNCHED DISC

PREPARED BY: *J. Mansfield*
J. W. Mansfield

3.1 Sample Size - Continued

	<u>MATERIAL</u>	<u>NUMBER OF PLIES</u>
Glass	(type)	
	108	16
	120	10
	128	8
	143	6
	341	6
	181	6
	182	4
	183	3
	184	3

<u>MATERIAL</u>	<u>NUMBER OF PLIES</u>
Asbestos	10
Carbon	4
Graphite	4
High Modulus Graphite (tape)	3

3.2 Number of determinations. Conduct one determination, on each sample, unless otherwise specified.

4. PROCEDURE

4.1 Temperature and pressure for resin type. Each resin type has unique flow properties which must be tested at temperatures and pressures peculiar to that type. Unless otherwise specified, use the following standard pressures and temperatures:

<u>RESIN TYPE</u>	<u>PRESSURE, PSI</u>	<u>TEMPERATURE, °F</u>
Phenolics		
Press Grade	500	325
Vacuum Grade	15	325
All Other	150	325
Epoxies (see 6.1)		
Unidirectional Tape	15	325
Vacuum Grade	15	275
All Other	50	275
Polyesters		
Press Grade	50	275
Vacuum Grade	15	275
Silicones	50	350



QUALITY ASSURANCE
TEST METHODS

DATE Revised

NUMBER PTM-19 G

April 28, 1975

Page 3 of 6

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY FLOW BY FLASH
REMOVAL OR PUNCHED DISC.

PREPARED BY: *J. Mansfield*
J. W. Mansfield

4.1 Continued

RESIN TYPE

PRESSURE, PSI

TEMPERATURE, °F

Other Resins

Must be specifically designated

All resin systems being utilized with the high modulus
graphite tapes

200

300

4.1.1 Tolerances:

(a) Pressure shall have a tolerance of $\pm 5\%$ but with a
minimum of ± 5 psi.

(b) Temperature shall have a tolerance of $\pm 5^\circ\text{F}$.

4.2 Lay-up for tapes. The following lay-up procedure shall be used
for tapes:

4.2.1 Fabric or mat tapes.

(a) Using strips about 5" long, lay up four (4) plies at
least 5" square. Lay up the plies so that the strips of each
successive ply are oriented 90° to the strips in the preceding
ply. Cut flow specimens using 4" x 4" template. Do not cut
specimens on the bias unless so designated.

4.2.2 Unidirectional tapes other than the high modulus graphite.

(a) Cut enough strips to make up four (4) plies 10" x 10".
Lay up the plies so that the fibers of each successive ply
are oriented 90° to the preceding ply.

(b) Using a 4" x 4" template, cut specimen on the bias.
Weigh each specimen to the nearest 0.01 gm.

(c) Sandwich the 4-ply specimen with two (2) 6" x 6" plies
of 181 glass fabric on each side and place between cellophane
release film. Proceed to 4.3 (b).

4.2.3 High modulus graphite unidirectional tapes.

(a) Cut three plies, 4" x 4" evenly spaced across the width
of the material and orient them 0° , 90° , 0° . Weigh the
layup to the nearest 0.01 gram.

(b) Sandwich the specimen with one 6" x 6" ply of TX-1040
release fabric, followed by two (2) 6" x 6" plies of Mochburg
bleeder fabric (each side). Place the entire layup between
cellophane or teflon release film. Proceed to 4.3 (b).



QUALITY ASSURANCE
TEST METHODS

DATE Revised
April 28, 1975

NUMBER PTM-19 G
Page 4 of 6

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY FLOW BY FLASH
REMOVAL OR PUNCHED DISC.

PREPARED BY: • *J. Mansfield*
J. W. Mansfield

4.3 Pressing Specimens

(a) If wet flow is to be determined, weigh the proper number of specimens to 0.01 gm. (see 6.2) or as specified. This weight is not necessary if dry flow is all that is desired.

(b) Properly align the specimens between release film (see 6.3). Position in the center of a press, preconditioned to the specified temperature, and close to the specified pressure as rapidly as possible.

(c) The time in the press shall be 10 minutes minimum but otherwise 3 ± 1 minutes greater than the gel time.

(d) Release pressure at end of time interval and immediately remove the release films if used. If dry flow is to be determined, use care in removing the release films so that none of the flash is lost.

4.4 Weighing the flash removal.

(a) If dry flow is to be determined, weigh the laminate with flash to 0.01 gm. or as specified.

(b) Scrape off the flash down to the original size with a dull knife, being careful to avoid removing any reinforcement from original dimensions.



QUALITY ASSURANCE
TEST METHODS

DATE
Revised
April 28, 1975

NUMBER PTM-19 G
Page 5 of 6

SUBJECT:

PHYSICAL TEST METHOD FOR:
WET AND DRY FLOW BY FLASH
REMOVAL OR PUNCHED DISC.

PREPARED BY:

J. Mansfield

J. W. Mansfield

4.4 (Continued)

(c) Reweigh the laminate to 0.01 gm. or as specified.

4.5 Punched disc.

If punched disc flow is to be determined, proceed as follows:

(a) Use a punch press to remove a 2-1/2 inch diameter disc from the center of the laminate.

(b) Weigh the disc to 0.001 gm. and measure the diameter of 0.01 inch.

5. CALCULATIONS AND REPORTING

5.1 Calculations. Calculate flow as follows:

5.1.1 Flow by flash removal.

5.1.1.1 Wet flow. Calculate wet flow as follows:

$$\text{Flow, Wt. \%} = \frac{W_1 - W_3}{W_1} \times 100$$

where: W_1 = Original weight, gms.

W_2 = Weight of laminate after pressing but before flash removal, gms.

W_3 = Weight after flash removal, gms.

5.1.1.2 Dry Flow. Calculate dry flow as follows:

$$\text{Flow, Wt. \%} = \frac{W_2 - W_3}{W_2} \times 100$$

where symbols are as specified in 5.1.1.1

5.1.2 Flow by punched disc.

5.1.2.1 Wet flow. Calculate wet flow as follows(see 6.4):

$$\text{Flow, Wt. \%} = \frac{W_1 - RW_4}{W_1} \times 100$$

where: W_1 = Original weight, gms.

W_4 = Weight of 2.50 inch disc, gms.

R = Ratio of area of 4" x 4" laminate to area of 2.50 inch disc

5.2 Reporting. Unless otherwise specified, report flow to three significant figures. Test results should be considered suspect if the range for triplicate determinations exceeds 2.5% for flows greater than 10%. For lower flows, tested at 150 psi or greater, the range of triplicate flows should not exceed 1.5%.



QUALITY ASSURANCE
TEST METHODS

DATE Revised
April 28, 1975

NUMBER PTM-19 G
Page 6 of 6

SUBJECT: PHYSICAL TEST METHOD FOR:
WET AND DRY FLOW BY FLASH
REMOVAL OR PUNCHED DISC.

PREPARED BY: *J. Mansfield*
J. W. Mansfield

6 NOTES

- 6.1 Pressure for epoxy systems. Use caution in selecting pressure and temperature for epoxy systems as a wide variety of applications require varied conditions.
- 6.2 Weighing. If greater accuracy is desired, weigh to 0.001 gm. In general, if the flow is 5% or less, weigh to nearest 0.001 gm.
- 6.3 Caul plates. Caul plates will not normally be used unless specifically designated.
- 6.4 Punched disc flow. Some specifications required flow to be calculated using dry weight in the denominators. This is not a standard calculation and cannot be defined as a wet or dry flow; however, the calculation is given below for reference:

$$\text{Flow} = \frac{W_1 - RW_4}{RW_4} \times 100$$

where symbols are as specified in 5.1.2.1



QUALITY CONTROL TEST METHODS

DATE

10-24 -78

NUMBER P.M.- 20 E

Page 1 of 2

SUBJECT:

PHYSICAL TEST METHOD FOR:
GEL TIME OF PREPREG MATERIALS

PREPARED BY:

James B. Rock
James B. Rock

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING

SALES

QUALITY ASSURANCE*

DATE N/A

DATE N/A

N/A

DATE

1. SCOPE

- 1.1 This method describes a procedure for the determination of gel time of impregnated material using a press probe.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- (a) Laboratory press, hydraulic
- (b) Balance, triple beam
- (c) Steel template, 2" x 2"
- (d) Laboratory knife and scraper
- (e) Timer, with 1 second intervals

ORIGINAL PAGE IS
OF POOR QUALITY

2.2 Materials

- (a) Release film, cellophane or nylon
- (b) Probes, wooden

3. SAMPLING

- 3.1 Sample size A representative sample of approximately 20 gms. will be required for each determination. Usually six inches by the width as supplied is sufficient for most materials.
- 3.2 Number of determinations Unless otherwise specified, single determinations per sample is sufficient.

4. PROCEDURE

- 4.1 Press preconditioning temperature Each resin system has its own unique properties and must be tested at temperatures peculiar to that type. Unless otherwise specified, precondition the press at the following temperatures: (all temperatures to be $\pm 5^\circ\text{F}$)

Resin TypeTemperature, °F

Epoxies

325

Phenolics

325

Polyesters And E760D Epoxy Prepregs

275

Silicones

350

Other resins

Must be specifically designated

* Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY CONTROL
TEST METHODS

DATE

10-24-78

NUMBER PTM-20E
Page 2 of 2

SUBJECT:

PHYSICAL TEST METHOD FOR:
GEL TIME OF PREPREG MATERIALS

PREPARED BY: •

James B. Rock

4.2 Preparation of Specimens

- (a) Using a steel template, randomly cut sufficient 2" x 2" plies to yield desired sample size. Normally 16 - 20 plies are sufficient.
- (b) Align the plies so that all cut specimens are evenly stacked atop each other.
- (c) Place a small piece of release film (1" x 1") on each side of the stack.

4.3 Gel Determination

- (a) Position previously prepared specimen in center of pre-conditioned press and close press as rapidly as possible and start timer at that time.
- (b) Adjust pressure according to flow in order to give a 1/4" bead of exuded resin around the sample.
- (c) Probe the bead with a wooden probe. Prior to gelation, a thread of resin can be draw with the probe. At this gelation point, the resin will not string and will appear to be of a jelly like consistency.
- (d) Drop the press at estimated gelation time, stopping the timer simultaneously.
- (e) Remove specimens quickly and inspect for signs of gelling by pulling off release film. Before this gelation point, the resin will adhere to the cellophane and will be soft and limp when bent, at the gelation point, the cellophane will generally release easily and the resin will be stiff when bending is attempted.
- (f) If it is felt that the material is not quite gelled, the test should be repeated.

NOTE: For S915/E760D for Thiokol, drop the press (d) after exuding resin and probe for gelation end-point.

5. CALCULATIONS AND REPORTING

5.1 Reporting

- (a) Report the gel time to the nearest second.
- (b) Test results should be considered suspect if repeated test differ by more than 5%.



QUALITY CONTROL
TEST METHODS

DATE

9/19/66

NUMBER PTM-29C

Page 1 of 3

SUBJECT: PHYSICAL TEST METHOD FOR:
SPECIFIC GRAVITY OF LIQUIDS, SEMI-
SOLIDS AND SOLIDS BY PYCNOMETER

PREPARED BY:*

P.D. McCart
P. D. McCart

9/20/66

P.D.

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING

SALES

QUALITY ASSURANCE*

DATE N/A

DATE N/A

J.P. Felt
DATE 9-22-66

1. SCOPE

1.1 Scope. This is a method for the determination of the specific gravity of liquids, semi-solids and solids which are neither water soluble nor appreciably absorb water at 25°C.

1.1.1 Method I. A procedure for liquids and materials that flow readily at 25°C.

1.1.2 Method II. A procedure for semi-solids and solids at 25°C.

1.2 Equivalent Method. The procedures described in Methods I and II are equivalent to ASTM-D1963-61.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Pycnometer, Gay-Lussac type
- (b) Pycnometer, Hubbard type
- (c) Analytical balance, minimum sensitivity 0.0001 gm.
- (d) Water bath, capable of maintaining $25 \pm 0.2^\circ\text{C}$
- (e) Thermometer, 74° to 79°F in 0.1°F intervals
- (f) Forceps, 6 inch
- (g) Vacuum chamber

2.2 Materials.

- (a) Distilled water, recently boiled
- (b) Lint free cloth
- (c) Abrasive paper, fine

3. SAMPLING

3.1 Sample size.

3.1.1 Method I. The sample should be large enough to fill the pycnometer for the required number of determinations.

3.1.2 Method II. For semi-solid materials there should be sufficient sample to fill the pycnometer one-half full for the required number of determinations. For solid materials there should be enough sample for the required number of specimens approximately $3/8 \times 3/8 \times 1$ inch, or an equivalent volume.

*Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY CONTROL
TEST METHODS

DATE

9/19/66

NUMBER PTM-29C

Page 2 of 3

SUBJECT: PHYSICAL TEST METHOD FOR:
SPECIFIC GRAVITY OF LIQUIDS, SEMI-
SOLIDS AND SOLIDS BY PYCNOMETER

PREPARED BY: *

P. D. McCart
P. D. McCart

9/20/66

pm

PND

3.2 Number of determinations. Unless otherwise specified, conduct two determinations on each sample.

4. PROCEDURE

4.1 Calibration. If the pycnometer needs cleaning, see 6.1 Calibrate the pycnometer as follows:

- (a) Bring the pycnometer to room temperature and weigh to 0.0001 gm. (W_1).
- (b) Fill the pycnometer (in such a manner as to prevent the entrapment of air bubbles) with cooled, just previously boiled, distilled water at a temperature of about 20°C. Insert the stopper, taking care that no bubbles are trapped.
- (c) Immerse in the water bath at $25 \pm 0.10^\circ\text{C}$ until constant temperature is attained (approximately 20 minutes).
- (d) Blot the meniscus of water in the bore so that it is flush with the surface of the stem.
- (e) Remove the pycnometer from the water bath and wipe dry with a clean, lint free cloth. Cool the pycnometer slightly to prevent loss of water through capillary and weigh to 0.0001 gm. (W_2).

4.2 Method I.

4.2.1 Materials with a viscosity of 40 stokes or less.

- (a) Fill a clean, dry Gay-Lussac type pycnometer with the sample, taking care to prevent the entrapment of air bubbles.
- (b) Proceed as described in 4.1 (c), (d), and (e) recording the weight as W_3 .

4.2.2 Material flows readily but viscosity is greater than 40 stokes.

- (a) Fill a clean, dry Hubbard type pycnometer with the material to be tested, taking care to prevent the entrapment of air bubbles.
- (b) Proceed as described in 4.1 (c), (d), and (e) recording the weight as W_3 .

4.3 Method II. This method is for semi-solids and solids at 25°C.

- (a) Place the sample in a clean, dry Hubbard type pycnometer and fill about one-half full. If the sample is a solid, use a specimen approximately $3/8 \times 3/8 \times 1$ inch or a sample of approximately this volume. Smooth the surface on molded or laminated specimens.
- (b) Bring the pycnometer and its contents to room temperature and then weigh with stopper to 0.0001 gm. (W_3).
- (c) Proceed as described in 4.1 (b) through (e), (if the sample is porous or powdered, see 6.2), recording the weight as W_4 .

5. CALCULATIONS AND REPORTING

5.1 Calculations



QUALITY ASSURANCE
TEST METHODS

DATE

9/19/66

NUMBER PTM-29C

Page 3 of 3

SUBJECT: PHYSICAL TEST METHOD FOR:
SPECIFIC GRAVITY OF LIQUIDS
SEMI-SOLIDS AND SOLIDS BY PYCNOMETER

PREPARED BY: *

P.D. McCart
P. D. McCart

9/20/66

P.D.

5.1.1 Calibration. Calculate the pycnometer constant as follows:

$$K = W_2 - W_1$$

where: K = pycnometer constant

W₁ = weight of pycnometer, gms.

W₂ = weight of pycnometer, plus water, gms.

W₃ = weight of pycnometer, plus material, gms.

W₄ = weight of pycnometer, plus material, plus water, gms

5.1.2 Method I. Calculate specific gravity as follows:

$$\text{Specific gravity} = \frac{W_3 - W_1}{K}$$

where symbols are as specified in 5.1.1

5.1.3 Method II. Calculate specific gravity as follows:

$$\text{Specific gravity} = \frac{W_3 - W_1}{W_3 + K - W_4}$$

where symbols are as specified in 5.1.1

6. NOTES

6.1 Cleaning. See the cleaning procedure outlined for viscosity tubes in PTM-26.

6.2 Powdered or porous materials. If the material is powdered or porous, add sufficient distilled water to cover the sample. The pycnometer with water and sample should then be placed in a vacuum chamber and the pressure reduced almost to the point of boiling the water, allow to remain at this point for approximately 20 minutes then proceed to 4.1 (b).

SPECIFIC GRAVITY BY PYCNOMETER (METHOD 1)

NASA 71108/71109 (Resin) - PTM-29C

Equipment

- Glass Pycnometer - Hubbard
- Analytical Balance - sensitivity 0.0001 gm
- Water bath - maintaining 25 ± 0.1 C

Procedure

- Duplicate determinations - two pycnometer per sample
- Record pycnometer number and sample number
- Clean, empty pycnometer is recorded as W_1
- Pycnometer filled with distilled water is W_2 (after ~20 mins in 25 C water bath)
- Dry pycnometer and fill until 1/4" from top, careful not to entrap air bubbles
- Place into water bath for thirty minutes and then record as W_3

- Calculate as follows: $\frac{W_3 - W_1}{K} = \text{SpG}$

$$K = W_2 - W_1$$

K - Pycnometer Constant

- All weights are taken to 0.0001 gm and with lids on
- Report average
- If air is trapped, place open Pycnometer in a small beaker of hot water. This will force the bubbles to the top quickly.
- Make sure Pycnometers are clean and dry from any sample over spill before weighing



Chemicals, Inc.

QUALITY ASSURANCE
TEST METHODS

DATE

3/22/66

NUMBER PTM-47B

Page 1 of 2

SUBJECT:

PHYSICAL TEST METHOD FOR:
GEL TIME BY OIL BATH

APPROVED

PREPARED BY: * P. D. McCart

P. D. McCart *Jm*

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING

SALES

QUALITY ASSURANCE*

DATE

N/A

DATE

N/A

DATE *H. m. Toellner*
4 April 1966

1. SCOPE

1.1 Scope. This method describes two procedures for the determination of gel time by immersing the sample in an oil bath.

1.1.1 Method I. This method is in general applicable to phenolic resins.

1.1.2 Method II. This method is in general applicable to silicone resins.

1.2 Equivalent methods. Method I is equivalent to ASTM-D1472-62. Method II is equivalent to the Dow Corning gel method for Dow Corning 2106 resin.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

(a) Jar, Pyrex, 6-inch diameter, 8-inch height, or similar container

(b) Stirrer, electrical with rheostat

(c) Test tubes; Method I, 13 mm x 120 mm; Method II, 25 mm x 100 mm

(d) Stirring rods: Method I, wire, .06 to .08 inch diameter with 0.30 to 0.40 inch loop perpendicular to stem; Method II, glass, 1/4-inch diameter with flattened paddle of 0.5 to 0.6 inch diameter at the end

(e) Silicone oil, Dow Corning F-1-D173, 100 cs or equivalent

(f) Clamps and ringstand

(g) Thermoregulator with range to 480°F, control-sensitive to $\pm 1^\circ\text{F}$

(h) Thermometer, 20° to 600°F, 1°F intervals

(i) Timer

(j) Balance, minimum sensitivity 0.1 gm.

(k) Heater, 250 W, immersion type

3. SAMPLING

3.1 Sample size. Method I requires 2 ± 0.1 gm. for each determination. Method II requires 12 ± 0.1 gm. for each determination.

3.2 Number of determinations. Unless otherwise specified, run duplicate determinations.

*Signatures necessary for all Quality Assurance Procedures and Instructions.



Chemicals, Inc.

QUALITY ASSURANCE
TEST METHODS

DATE

3/22/66

NUMBER PTM-47B

Page 2 of 2

SUBJECT:

PHYSICAL TEST METHOD FOR:
GEL TIME BY OIL BATH

APPROVED

PREPARED BY: P. D. McCart

P. D. McCart *Jm*

4. PROCEDURE

4.1 Preheating bath. Preheat the oil bath to $338^{\circ} + 1.5^{\circ}\text{F}$ for Method I or to $480^{\circ} + 2^{\circ}\text{F}$ for Method II. If necessary, add oil to bring the oil level to within 1 inch of the top. Using an electric stirrer, stir bath thoroughly and continually for at least 5 minutes prior to testing and throughout the test to assure even heat dissipation.

4.2 Method I.

4.2.1 Weighing sample. Weigh a $2 + 0.1$ gm. sample into a test tube. Place the wire stirrer, looped end down, into the test tube.

4.2.2 Immersion. Lower the test tube into the holder. The test tube holder is to be sized to hold the tube loosely suspended by the rim and shall be positioned so that the rim of the test tube is $1.5 + 0.25$ inches above the surface of the oil. The stopwatch is started immediately upon immersion.

4.2.3 Stirring sample. Agitate the resin with rapid vertical strokes, not more than $1/4$ inch for the first 40 seconds. After 40 seconds, agitate at the rate of 4 strokes every 5 seconds.

4.2.4 End point. The end point is defined as that time when there is no motion of the test tube in relation to the stirrer, i.e. when the tube moves up and down with the stirrer. CAUTION: Be sure the holder is loose enough to permit this.

4.3 Method II.

4.3.1 Weighing sample. Weigh a $12 + 0.1$ gm. sample into a test tube. Place the glass rod, paddle down, into the test tube.

4.3.2 Immersion. Lower the test tube into the holder so that the rim of the test tube is $1.5 + .25$ inches above the surface of the oil. Tighten the holder to prevent slippage of the test tube. The stopwatch is started immediately upon immersion.

4.3.3 Stirring sample. Stir the sample by twisting the glass rod. Do not lift the rod up and down. Continue turning the paddle for three minutes.

4.3.4 End point. The end point is defined as that time when the glass rod can be turned 90° and will spring back considerably upon releasing.

5. CALCULATIONS AND REPORTING

5.1 Unless otherwise specified, report the average of two tests in minutes and seconds to the nearest second. Test results should be considered suspect if the range for duplicate determinations exceeds 10 seconds for gel times less than 6 minutes or 20 seconds for gel times greater than 6 minutes.



QUALITY ASSURANCE
TEST METHODS

DATE
12/21/81

NUMBER
PTM-61B
Page 1 of 2

SUBJECT:

PHYSICAL TEST METHOD FOR:
LINEAR THERMAL EXPANSION

PREPARED BY:
F. Bancroft

1. SCOPE

1.1 Scope. This is a method for the determination of linear thermal expansion using the differential scanning calorimeter. The thermal expansion of plastic is reversible. Upon it are superimposed changes in length due to changes in moisture content, degree of polymerization, loss of plasticizer or solvents, release of stresses or other factors which are mainly irreversible. This method of test is intended to yield the linear thermal expansion under exclusion of these accidental factors. In general, it will not be possible to exclude all of these spurious factors and thus the method can be expected to give only approximate values. This method is an adaptation of ASTM E228-71.

2.0 EQUIPMENT AND MATERIALS

2.1 Equipment

- A. Differential Scanning Calorimeter
- B. DSC User's Manual
- C. Helium Gas source

3.0 SAMPLING

- 3.1 Preparation. All samples shall be cut or shaped from moldings, castings, or laminates by methods under conditions that give a minimum of strains or molecular anisotropy.
- 3.2 Dimensions. Samples shall be 1/4 by 1/4 by 1/4 inches with the ends machined flat and parallel.

4.0 PROCEDURE

- A. Calibrate the DSC as per user's Manual
- B. Turn on the helium atmosphere or other gas as specified
- C. Set approximate upper and lower temperatures
- D. Measure sample height, width, and thickness to nearest 0.0001 inch
- E. Place sample into the TMS (DSC) apparatus, noting the ply direction
- F. Zero out the chart
- G. Raise temperature to upper limit at 40°F per minute. Hold upper limit for 5 minutes.
- H. Cool sample at 40°F per minute until lower limit is reached. Hold at lower limit for 5 minutes.
- I. Repeat steps G and H three (3) times. Disregard the first cycle of heating and cooling. This was done to condition the sample.
- J. Measure the temperature difference at the end of each heating or cooling period.



QUALITY ASSURANCE
TEST METHOD

DATE 12/21/81

NUMBER PTM-61B
Page 2 of 2

SUBJECT:

PHYSICAL TEST METHOD FOR:
LINEAR THERMAL EXPANSION

PREPARED BY:

F. Bancroft

- K. Measure the shrinkage difference at the end of the heating and cooling periods within each cycle.

5.0 CALCULATIONS AND REPORTING

- 5.1 Calculations; Calculate the coefficient of linear thermal expansion as follows:

$$E = \frac{\Delta L}{L \Delta T}$$

Where: E = coefficient of thermal expansion, in//in./°F x 10⁻⁶
L = test length of specimen at room temp., inch
T = temperature difference, °F, over which the change in length was measured
 ΔL = Average of change in length due to heating and cooling



QUALITY ASSURANCE
TEST METHODS

DATE
12/9/66

NUMBER PTM-71B
Page 1 of 2

SUBJECT: PHYSICAL TEST METHOD FOR:
ASH CONTENT OF FILLERS
AND RELATED MATERIALS

PREPARED BY: *

P.D. McCart
P. D. McCart 21 DEC 66

P.N.D.
Jm

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING

SALES

QUALITY ASSURANCE*

DATE N/A

DATE N/A

J. F. Galt
DATE 1-4-67

1. SCOPE

1.1 Scope. This method describes a procedure for the determination of ash content of ignitable fillers, pigments and related materials which will ash at 1750°F.

1.2 Equivalent procedures. This procedure is equivalent to ASTM-D1506-59.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

(a) Muffle furnace

(b) Oven, forced air

(c) Analytical balance, minimum sensitivity, 0.1 mg.

(d) Crucibles, porcelain, wide form with covers

3. SAMPLING

3.1 Sample size. Obtain a representative sample of at least 5 grams.

3.2 Number of determinations. Unless otherwise specified, run duplicate determinations.

4. PROCEDURE

4.1 Drying sample. Condition about 5 grams of sample in a porcelain crucible in a forced air oven for 60 + 5 minutes at 225 + 5°F.

4.2 Weighing. Weigh about 2 grams to the nearest 0.0001 gram into a prefired and weighed porcelain crucible.

4.3 Ignition. Place sample into muffle furnace at 1750 + 50°F. Ignite for about 4 hours. A longer time period may be necessary. Keep the furnace door open about 1/4 inch to admit air. Check for completeness of ashing by cooling the sample in a desiccator, weighing, igniting for 1 hour, then cooling in a desiccator and re-weighing.

4.4 Final weight. When ashing is determined to be complete, cool the sample in a desiccator, then reweigh crucible and residue to 0.0001 gm.

* Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY ASSURANCE
TEST METHODS

DATE

12/9/66

NUMBER

PTM-71B

Page 2 of 2

SUBJECT:

PHYSICAL TEST METHOD FOR:
ASH CONTENT OF FILLERS
AND RELATED MATERIALS

PREPARED BY: *

P.D. McCart
P. D. McCart 21 DEC 66

PND
(Signature)

5. CALCULATIONS AND REPORTING

5.1 Calculations. Calculate ash content as follows:

$$\text{Ash Content, Wt. \%} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

where: W_1 = weight of prefired crucible, gms.
 W_2 = weight of crucible plus sample, gms.
 W_3 = weight of crucible plus residue, gms.

5.2 Reporting. Unless otherwise specified, round off and report ash content to 0.01%. Duplicate determinations should be considered suspect if they differ by more than 0.1% ash content.

SUBJECT: PHYSICAL TEST METHOD FOR:
MINIMUM TACK TEMPERATURE FOR
PATTERN GRADE PREPREG

APPROVED
PREPARED BY: * P. D. McCart


APPROVAL SIGNATURES:

GENERAL MANAGER	ENGINEERING	PRODUCTION	PRODUCTION CONTROL
DATE N/A	DATE N/A	DATE N/A	DATE N/A
PURCHASING	SALES		QUALITY ASSURANCE*
DATE N/A	DATE N/A		<i>H. M. Toellner</i> DATE 14 March 1966

1. SCOPE

- 1.1 Scope. This method describes a procedure for establishing the minimum tack temperature of prepreg materials by lap shear where the specimen overlap is under a specified pressure while heating. This method does not give a shear strength.
- 1.2 Purpose. The purpose of this method is to confirm blocking temperature of pattern grade material.

2. EQUIPMENT AND MATERIALS
2.1 Equipment.

- (a) Asbestos laminates, 3"x3"
- (b) Hot plate, variable temperature control
- (c) Metal block, 2" x 2" x 1/4", 125 ± 5 grams
- (d) Press device, capable of applying 50 ± 2 lbs. of force
- (e) Stop watch and/or electric timer
- (f) Weight, 15 lbs. (or other specified weight)

2.2 Materials.

- (a) Cellophane

3. SAMPLING

- 3.1 The sample should be large enough to provide a sufficient number of 1" x 3" specimens to determine minimum tack temperature. Unless otherwise specified, specimens shall be cut so as to optimize sample size and shall be in either the warp or fill direction.

4. PROCEDURE
4.1 Preparation of test specimen.

- 4.1.1 Preconditioning hot plate. Unless otherwise specified, preheat the hot plate to the specified temperature (+ 3°F). Place the 2" x 2" x 1/4" block and the 15-pound (or other specified) weight on the hot plate during this preheat time.
- 4.1.2 Heating test specimen. Take two 1" x 3" specimens and form a one-inch overlap, placing warp side to fill side. Place between a fold of

*Signatures necessary for all Quality Assurance Procedures and Instructions.

SUBJECT: PHYSICAL TEST METHOD FOR:
MINIMUM TACK TEMPERATURE FOR
PATTERN GRADE PREPREG

APPROVED

PREPARED BY: P. D. McCart *J.P.H.*

cellophane. Place the cellophane-wrapped specimen on the hot plate, place the 2" x 2" x 1/4" metal block on the overlap, and place the 15-pound (or other specified) weight on the 2" x 2" x 1/4" metal block. Allow the specimen to heat for 30 ± 2 seconds. Remove the weight and metal block and take specimen from the hot plate.

4.1.3 Specimen does not bond. If the specimen has not bonded, confirm this condition with an additional specimen before changing temperature.

4.1.4 Specimen is bonded. As quickly and delicately as possible, (5 seconds maximum), place the overlapped area between the platens of a room temperature press. The platens of the press shall be covered with asbestos laminates which are covered with cellophane. Unless otherwise specified, apply 50 ± 2 lbs. until the specimen is cool (about 1 to 2 minutes).

4.2 Testing specimen. Clamp one end of the specimen in jaws of spring load dial scale. Load other end of the specimen at a constant rate thus forming a lap shear test. Continue loading until shear or break.

4.3 Minimum tack temperature.

4.3.1 Specimen supports weight. If the specimen supports a measurable load (see 6.1), decrease temperature (usually at 10°F intervals) until specimen will not support a measurable load.

4.3.2 Specimen does not support weight. If the specimen does not support a measurable load, continue testing, increasing the hot plate temperature (usually at 10°F intervals) until the specimen will support a measurable load. Report load in pounds. Test two additional specimens at this temperature.

5. CALCULATIONS AND REPORTING

5.1 Reporting.

(a) Report the average load in pounds of three specimens at minimum tack temperature.

(b) Report minimum tack temperature at which a measurable load is supported. The minimum tack temperature must be established by a minimum of three specimens.

6. NOTES

6.1 Measurable load. A measurable load for the purpose of this test shall not be less than three pounds. The measurable load shall be as designated. When not designated, it shall be five pounds.



QUALITY ASSURANCE
TEST METHODS

DATE

4/27/67

NUMBER PTM-84

Page 1 of 3

SUBJECT: PHYSICAL TEST METHOD FOR:
SPECIFIC GRAVITY OF SOLIDS BY
"AIR COMPARISON" PYCNOMETER

PREPARED BY:*

P. H. Davis 4/28/67

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING

SALES

QUALITY ASSURANCE*

DATE N/A

DATE N/A

DATE 5-17-67

1. SCOPE

- 1.1 This procedure describes a method for determination of apparent volume of raw fabrics, prepregs, cured parts and other related materials by use of an air comparison pycnometer. After apparent volume has been measured, apparent density can be calculated. This method is suitable for the analysis of non-surface active, non-compressible materials (see 6.1).

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

- (a) Pycnometer, Model 930, Beckman, or equivalent
- (b) Oven, vacuum
- (c) Balance, analytical, minimum sensitivity 0.001 gm.
- (d) Gas bottle with regulator (see 6.2)
- (e) Vacuum source
- (f) Laboratory knife

3. SAMPLING

- 3.1 Sample size. Obtain a representative size sample of approximately five (5) grams for bulky materials (raw fabrics should be about 1" by 36"). For solid materials (cured parts), a sample size up to 50 c.c. can be measured. A greater degree of accuracy can be achieved by using larger sample sizes, but the sample size will be dependent upon the ability of the sample to fit into the sample cup (maximum volume 50 c.c.).
- 3.2 Number of determinations. Unless otherwise specified, one determination will suffice but, multiple measurements must be made (see paragraph 4.3.1).

4. PROCEDURE

- 4.1 Sample conditioning. Unless otherwise specified, condition sample by inserting in a vacuum oven heated to 275°F. After exposure under full vacuum for 20 to 30 minutes, remove sample from oven and immediately weigh to nearest 0.001 gram.

* Signatures necessary for all Quality Assurance Procedures and Instructions.



QUALITY ASSURANCE
TEST METHODS

DATE

4/27/67

NUMBER

PTM-84

Page 2 of 3

SUBJECT: PHYSICAL TEST METHOD FOR:
SPECIFIC GRAVITY OF SOLIDS BY
"AIR COMPARISON" PYCNOMETER

PREPARED BY: *

P. H. Davis *m. d. Jan*
P. H. Davis 4/28/67

4.2 Instrument calibration.

- 4.2.1 Initial warm-up. At the start of a series of measurements the instrument should be loosened and warmed-up. This is done by turning both the measuring and reference handwheels in and out several times with the purge and coupling valves open.
- 4.2.2 Volume check. Accuracy of the unit should be checked for volume by inserting either the 1" or 1-1/2" steel test ball in the sample cup, and measuring volume of the ball per paragraph 4.3. The volume readings obtained from the test balls should be a consistent 8.58 c.c. for the 1" steel ball and 28.96 c.c. for the 1-1/2" steel ball (see 6.3). If the ball measurement exceeds a volume tolerance of + .15 c.c. for either ball, refer to the Instruction Manual for adjustment instructions.
- 4.2.3 Zero check. The zero check procedure is identical to paragraph 4.3, but is made with the sample cup clean and empty. If the final counter reading is other than zero, refer to the Instruction Manual for adjustment instructions.

4.3 Specimen volume measurements. Unless otherwise designated, the following procedure shall be utilized in determination of apparent volume:

- (a) Adjust gas pressure regulator on the helium tank to a pressure no greater than 2 psi. Turn vacuum supply on.
- (b) With purge and coupling valves open, rotate both handwheels in a counter-clockwise direction to the extreme position. Turn the measuring wheel until starting number is reached (located on a plate affixed to the side of the case above the measuring handwheel).
- (c) Insert previously conditioned and weighed specimen into the sample cup and insert sample cup into position and secure it firmly.
- (d) Open purge and coupling valves.
- (e) Open vacuum valve and allow 10 seconds for system to evacuate, then close valve.
- (f) Open gas (helium) valve and allow 5 seconds for pressure equilibrium, then close gas valve.
- (g) Open vent valve for 5 seconds to allow for pressure equilibrium, then close vent and purge valves.
- (h) Wait for 10 seconds then loosen the coupling valve (this should be rotated a couple of times).
- (i) Wait for 10 seconds then, turn both handwheels clockwise simultaneously until the reference wheel stops. Apply a minimum amount of pressure after the wheel initially stops. Keep the pointer on the scale during this process.
- (j) Wait for 10 seconds and adjust pointer by image alignment to the zero mark with the measuring handwheel.
- (k) Open coupling valve. Read specimen volume on counter directly in c.c.



QUALITY ASSURANCE
TEST METHODS

DATE

4/27/67

NUMBER

PTM-84

Page 3 of 3

SUBJECT: PHYSICAL TEST METHOD FOR:
SPECIFIC GRAVITY OF SOLIDS BY
"AIR COMPARISON" PYCNOMETER

PREPARED BY: *

P. H. Davis

M.O.

(Signature)

P.H. Davis 4/28/67

4.3 Cont'd.

(1) Open purge valve and vent valve, then close vent valve and repeat steps (b) through (k) until consistent readings are obtained on each specimen.

NOTE: When opening coupling valve, observe null (differential pressure) indicator. If it shifts position, true null was not obtained and the run should be repeated.

5. CALCULATIONS AND REPORTING

5.1 Calculations. To calculate apparent density (see 6.4) from apparent volume use the following formula:

$$\text{Density} = \frac{\text{Weight of dried specimen, gms.}}{\text{Volume reading obtained, c.c.}}$$

5.2 Reporting. Report specific gravity to four significant figures. In addition to reporting specific gravity, report specimen weight and apparent volume.

6. NOTES

6.1 Instrument limitations. Materials such as carbon fabric will not yield reproducible values however graphite fabric will yield reasonably accurate results even though it is surface active.

6.2 Usable gases. Any inert gas may be used. Unless otherwise specified use helium.

6.3 Ball checks. When checking several samples during one series of tests, periodic checks of the ball should be made to make sure the instrument is still reading accurately. This is best done by checking the ball in between every third or fourth sample that is being tested.

6.4 Specific gravity. Density and specific gravity may be considered identical for all practical purposes in the measurements of materials whose density do not vary appreciably with temperature.

SPECIFIC GRAVITY

NASA 71108/71109 (fabric) per PTM-84

Equipment

- Quantachrome Stereopycnometer

Sampling

- 5 - 10 mg - cut into 1-1/2" x 1-1/2" squares to fit easily into sample cup

Procedure

- Equipment warm-up; purge for 10 - 15 minutes with cell and vent valves open
- Record weight of sample cup
- Record weight of sample and sample cup
- subtract for weight of sample
- Insert into chamber; close and bring digital display to zero
- Close cell vent and turn to VA out
- With flow valve open; pressurize to slightly less than 20 psi
- Close flow valve and stabilized reading is recorded as P
- Turn to VA in and when stabilized reading is attained; this is P
- Vent pressure slowly
- Calculate volume
- Do three determinations; then report average
- Calibration is done prior to the beginning of testing
- If pressure does not stabilize, check sample chamber for proper sealing and check pressure on helium tank



QUALITY ASSURANCE
TEST METHODS

DATE

10/9/74

NUMBER

PTM-98
Page 1 of 2

SUBJECT: PHYSICAL TEST METHOD FOR DETERMINATION OF
PRESS LAMINATE RESIDUAL VOLATILES

PREPARED BY: *

G. Tulloch

APPROVAL SIGNATURES:

GENERAL MANAGER

ENGINEERING

PRODUCTION

PRODUCTION CONTROL

DATE N/A

DATE N/A

DATE N/A

DATE N/A

PURCHASING

SALES

QUALITY ASSURANCE*

DATE N/A

DATE N/A

John M. Maffei
DATE 10-10-74

1.0 SCOPE

1.1 Method for determining residual volatile in cured laminates.

2.0 EQUIPMENT AND MATERIALS

2.1 Equipment

- a) Laboratory forced air oven, 325°F capability
- b) Laboratory press, hydraulic, 16,000 lbs., 325°F capability
- c) Analytical balance
- d) 4" X 4" steel template
- e) Laboratory knife
- f) Carbide bladed table saw or hack saw

2.2 Materials

- a) Teflon coated glass cloth release fabric

3.0 SAMPLING

3.1 Sample size: The sample shall be large enough so that the required number of 4" X 4" plies, cut on the bias, may be obtained to make a laminate 0.250" thick.

4.0 PROCEDURE

4.1 Laminate: Using 4" X 4" template, cut required number of plies on the bias. Stack and load in 325°F press between Teflon coated glass cloth release material. Apply contact pressure for 30 seconds, dump press; repeat twice and apply 1,000 psi for four hours at 325°F. Remove laminate from press, remove flash with knife, and cut three 1" X 1" pieces from laminate as follows: (Refer to Figure 1)

- a) Cut 1" X 4" center strip parallel to two outer edges.
- b) Cut three 1" X 1" pieces from this strip, starting at one end without trimming outside edge.
- c) Desiccate pieces.

SUBJECT: PHYSICAL TEST METHOD FOR DETERMINATION OF
PRESS LAMINATE RESIDUAL VOLATILES

PREPARED BY: *

G. Tulloch

4.2 Test for Residual Volatiles: Each sample described above, after desiccation for a minimum of 18 hours, is weighed. This is sample weight. Samples are placed in forced air oven for four hours at 325°F, removed and weighed hot. Four-hour weight loss is calculated:

$$\text{Residual Volatile of each sample} = \frac{\text{Weight Loss}}{\text{Sample Weight}} \times 100\%$$

Residual Volatile of material = Arithmetic Average of
above three samples

4.3 Tolerances:

0.250" Laminate Thickness, $\pm 5\%$
325°F Press, $\pm 15^\circ\text{F}$
1,000 psi Press, $\pm 5\%$
325°F Oven, $\pm 10^\circ\text{F}$

5.0 REPORTING

5.1 Report Residual Volatile to 2 significant figures.

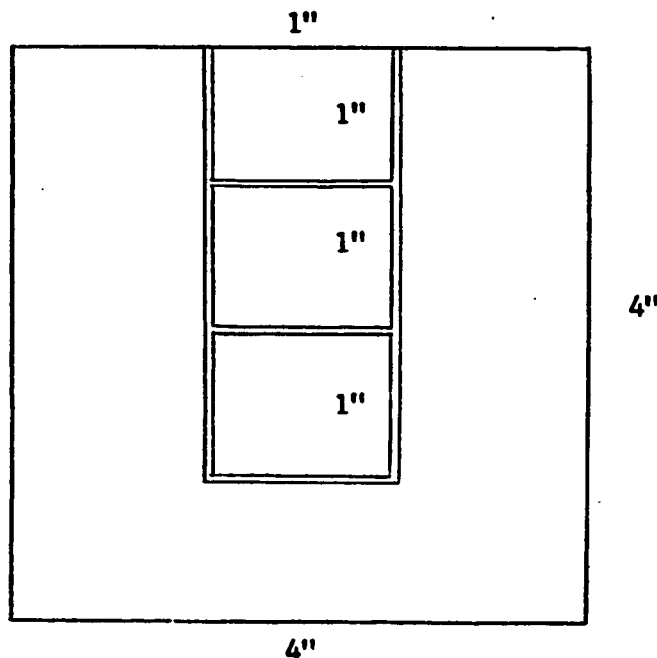


FIGURE 1

TABLE OF CONTENTS
MISCELLANEOUS TEST METHODS

NAS8-36298

U.S. POLYMERIC O.E. 36298

HITCO

MDQAI 5070	Determination of Tack of Uncured Preimpregnated Tapes and Broadgoods
MDQAI 5560	Determination of Carbon Content
-----	SEM Test Method for Carbon Filler

(NOTE: Individual pages are not numbered except in sequence for
each test method.)

CITED BUT NOT INCLUDED

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM D 792	Specific Gravity and Density of Plastics by Displacement
ASTM D 1512	Carbon Black - pH Value
ASTM D 1682	Breaking Load and Elongation of Textile Fabrics
ASTM D 2583	Indentation Hardness of Rigid Plastics by Means of a BARCOL Impressor

FEDERAL TEST METHOD STANDARD NO. 406

1011	Tensile Properties of Plastics
1021	Compressive Properties of Rigid Plastics
1031	Flexural Properties of Plastics
1041	Shear Strength (Double Shear)

H I T C O
Gardena, California

NO. 5070

PAGE 1 OF 2

DATE 11-21-68

REV. _____

REV. BY _____

QUALITY ASSURANCE
I N S T R U C T I O N

1.0	<u>DEPARTMENT:</u>	Quality Assurance Process Control Laboratory
2.0	<u>SUBJECT:</u>	Determination of Tack of uncured preimpregnated tapes and broadgoods
3.0	<u>PURPOSE:</u>	To furnish instructions for laboratory personnel to follow in the testing of material
4.0	<u>SCOPE:</u>	This instruction describes the method for determination of tacking properties of uncured preimpregnated tapes and broadgoods

PREPARED BY: _____

W. H. Boarder

APPROVED BY: _____

Quality Assurance Manager

5.0 EQUIPMENT

- (A) Press with a 6" x 6" platen
- (B) 2-Caul sheets
- (C) Universal testing machine
- (D) Instron pneumatic jaws with rubber facings
- (E) Stanley knife

6.0 PROCEDURE

- 6.1 Cut 6 strips 1" x 4" from each sample.
- 6.2 Preheat press and caul sheets to 200 \pm 5°F.
- 6.3 Place the strips between the caul sheets with a 1.0 inch overlap and place into the press.
- 6.4 Close press to 50 psi as rapidly as possible and hold this pressure for 60 seconds.
- 6.5 Remove caul sheets from the press and gently remove the laminated tape or broadgood. Place the sample between 2-1/8" thick caul sheets and allow to cool to room temperature.
- 6.6 Adjust the universal testing machine to provide a 3 inch gage length between grips.
- 6.7 Align specimen in top grip and close grip.
- 6.8 Align specimen in bottom grip to permit equal application of tension and close bottom grip.
- 6.9 Apply the tension load of a cross heat speed of 0.05 inches per minute until specimen breaks

7.0 CALCULATIONS

Record the load in lbs per inch and the time in seconds. Report the average of the 3 specimens.

HITCO
MATERIALS DIVISION
GARDENA, CALIFORNIA

MATERIALS DIVISION
QUALITY ASSURANCE INSTRUCTIONS

MDQAI 5560

REVISION A

DETERMINATION
OF
CARBON CONTENT

MANUFACTURING N/A

ENGINEERING Harry Shepherd

QUALITY ASSURANCE J. M. M. M. M.

LABORATORY John H. J. J. J.

MARKETING N/A

DIVISION MANAGER J. H. J. J. J.

REVISION DATE 1 July 85

MATERIALS DIVISION QUALITY ASSURANCE INSTRUCTIONS

MDQAI 5560

DEPARTMENT: Quality Assurance And Process Laboratory, Dept. 20

SUBJECT: Sample preparation and operation instructions for the LECO CHN-600 Carbon, Hydrogen, Nitrogen Analyzer

PURPOSE: To furnish instruction for laboratory personnel in the preparation, analysis, and report of data for content of carbon in high carbon content materials and composites.

SCOPE: This instruction provides a method for the analysis of carbon expressed as weight percent.

REVISION

CHANGE(S)

A Virtually the complete instruction has been rewritten. Most paragraphs have been renumbered to reflect actual instrument operational sequences. Blanks, calibrations, and data stack manipulations are required to optimize instrument accuracy and precision. Specimen preparation is modified to yield data relative to discreet web locations.

MATERIALS DIVISION QUALITY ASSURANCE INSTRUCTIONS

MDQAI 5560

1.0 EQUIPMENT

- 1.1 LECO CHN-600 Carbon, Hydrogen, Nitrogen Analyzer.
- 1.2 #1 stopper borer.
- 1.3 Large tin capsules, LECO Part Number 502-0404.
- 1.4 Compressed air, clean, with dew point of at least -70°F.
- 1.5 O₂, 99.99%.
- 1.6 CO₂, 99.9%.
- 1.7 Graphite standard, 99.99% carbon content, with particle size of 325 mesh or finer.

2.0 INSTRUMENT PREPARATION

2.1 Furnace Temperature.

- 2.1.1 Primary and secondary furnace temperatures must be gradually increased to the analytical set point of 950°C. Press SYSTEM UPDATE.
- 2.1.2 Press NO for each question until "Security Area" appears on the display. Press YES and enter the security code. Press ENTER.
- 2.1.3 Press NO for each question until "Set Temperature" appears on the display. Press YES.
- 2.1.4 Both primary and secondary furnace temperatures will be set at 0600 (600°C). Enter primary temperature as 0700. Press ENTER. Enter secondary temperature as 0700. Press ENTER.
CAUTION: Do not change the oven temperature.
- 2.1.5 Every ten to fifteen minutes repeat 2.1.1 thru 2.1.4 to raise the furnace temperatures in 100°C increments to a set point of 950°C.

2.2 Crucible Change.

- 2.2.1 The ceramic crucible, inside of the U-tube, must be changed after every thirty (30) analyses. The wad of quartz wool that supports the crucible must be changed daily.
- 2.2.2 Refer to page 45 of the instrument Instruction Manual for the specific procedure to change the crucible and quartz wool.

3.0 SPECIMEN PREPARATION

3.1 Textiles — Raw Fabric or Prepreg.

- 3.1.1 Cut three specimens, 3" x 7" nominally, from the sample as illustrated in Figure 1.
- 3.1.2 Place the specimens in a convection oven and dry at 125 ± 5°C for a minimum of two (2) hours.

MATERIALS DIVISION QUALITY ASSURANCE INSTRUCTIONS

MDQAI 5560

3.0 SPECIMEN PREPARATION (cont.)

- 3.1.3 Remove the specimens from the oven and dessicate to ambient temperature.
- 3.1.4 Fold the specimens in half and then fold again in thirds as illustrated in Figure 2.
- 3.1.5 With a sharp #1 stopper borer, hole the folded specimen six times as illustrated in Figure 3.
- 3.1.6 Insert the borer in to the top of a tared tin capsule and use the bore cleaning tool to gently push the material out of the borer.
- 3.1.7 Crimp the top of the capsule with forceps as the borer is withdrawn to prevent escape of sample material.
- 3.1.8 Weigh the sample capsule and subtract this weight from the capsule tare weight to obtain the net sample weight. Normal instrument operation requires a net sample weight of $0.10 \pm 0.01g$.

3.2 Solids — Cured Composites or Powders.

- 3.2.1 Cured composites shall be milled or cut to dimensions of approximately 1/8th inch square by 1/2 inch.
- 3.2.2 The sample may then be loaded into a tin capsule with forceps and weight determination made per 3.1.8.
- 3.2.3 At the discretion of the Laboratory Supervisor, the sample may be ground to a particle size of less than 60 mesh and mixed for homogeneity.
 - 3.2.3.1 The sample may then be loaded into a tin capsule with use of the funnel on the LB-20 analytical balance.

4.0 INSTRUMENT CALIBRATION

4.1 Blank Determination.

- 4.1.1 Perform three to five blank determinations following introduction of a new ceramic crucible.
- 4.1.2 Refer to page 25 of the instrument Instruction Manual for specifics of blank operational conditions.

4.2 Calibration By Burns.

- 4.2.1 A 99.99% carbon content standard, ground to 325 mesh or finer, shall be used as a carbon standard.
- 4.2.2 Use the funnel attachment on the LB-20 analytical balance to load carbon standard in to a tared tin capsule. Normal operation requires a net carbon standard weight of $0.1 \pm 0.01g$.
- 4.2.3 A carbon standard shall be run, analytically, after every nine (9) sample analyses. This will yield three standard determinations per calibration stack.

MATERIALS DIVISION QUALITY ASSURANCE INSTRUCTIONS

MDQAI 5560

4.0 INSTRUMENT CALIBRATION (cont.)

4.3 Calibration By Gas Dose.

- 4.3.1 At the discretion of the Laboratory Supervisor, calibration may be performed by gas dose.
- 4.3.2 Three (3) gas dose calibrations shall be run with seven (7) doses per calibration.
- 4.3.3 Refer to page 31 of the instrument Instruction Manual for operational specifics.

5.0 ANALYTICAL OPERATION

- 5.1 Press ID CODE and enter the sample identification (up to eight (8) digits). Press ENTER.
- 5.2 Press MANUAL WEIGHT and enter the net sample weight. Press ENTER.
- 5.3 Load the capsule, with sample, in the appropriate position.
- 5.4 Press ANALYZE to begin the analytical routine.
- 5.5 Further samples and weights can be entered (up to thirty (30)) while the analytical routine is in progress.
- 5.6 Each analytical result may be printed, at the option of the operator, as determined or only retained in the instrument data stack.

6.0 CALIBRATION OF DATA STACK

- 6.1 On completion of the last analysis the entire data stack must be calibrated by burns. This assures optimal instrument response.
- 6.2 Press STANDARD CHECK. The console will display "CALIBRATE BY BURNS YES/NO". Press YES.
- 6.3 "DISPLAY AS DET. [detected] ANSWERS YES/NO" will appear on the console display. Press YES. The current calibration stack values will then be printed.
- 6.4 "CALIBRATE CARBON YES/NO" will appear. Press YES.
- 6.5 "CARBON STD. AS DET. ##.## MOD. [modify] BY KBD [keyboard]" will appear. Enter 99.99.
- 6.6 The instrument will individually list the memory stack and ask whether or not to include that displayed determination as a standard in the calibration process. Press YES for each standard as it is displayed, otherwise press NO.
- 6.7 After the last entry is displayed, the new calibration value will be calculated and the recalculated answers will be printed.

7.0 REPORT

- 7.1 Unless otherwise specified, report the average of three determinations.
- 7.2 Unless otherwise specified, report values shall be to the nearest 0.1 %.

MATERIALS DIVISION QUALITY ASSURANCE INSTRUCTIONS

MDQAI 5560

8.0 INSTRUMENT IDLE

- 8.1 Should instrument operation not be required for more than eight (8) hours, such as over night or weekend idle, the primary and secondary furnace temperatures shall be reduced to preserve the U-tube and furnace reagent.
- 8.2 Press SYSTEM UPDATE. Press NO for each question until "SECURITY AREA" appears. Press YES.
- 8.3 Enter the security code. Press ENTER.
- 8.4 Enter the primary furnace temperature as 0600 (600°C). Press ENTER. Enter the secondary furnace temperature as 0600. Press ENTER. Press ENTER to maintain the oven at the preset temperature.
- 8.5 Press GAS to turn off the instrument gas flow.

MATERIALS DIVISION QUALITY ASSURANCE INSTRUCTIONS

MDQAI 5560

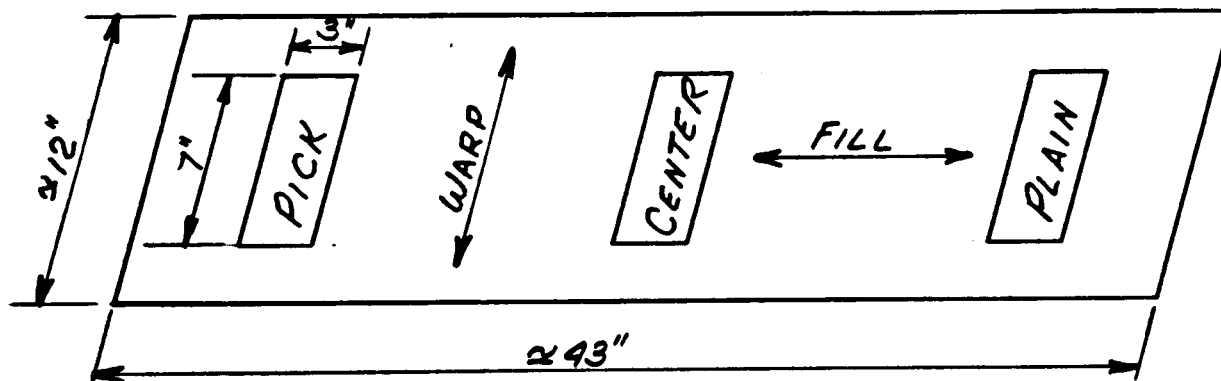


FIG. 1

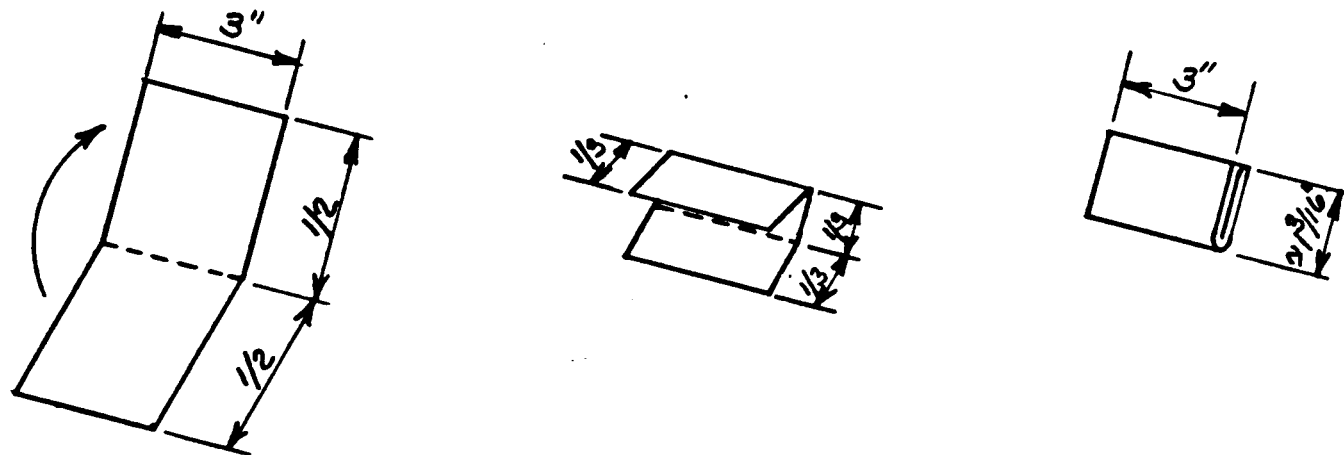


FIG. 2

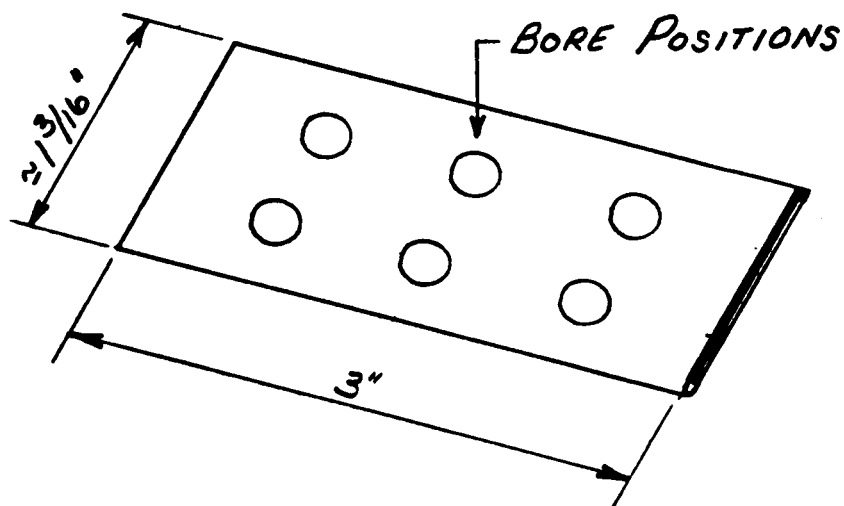


FIG. 3



MICROGRAPHICS

1672 KAISER AVENUE

IRVINE,

CALIFORNIA

92714

(714) 250-4544

Dec. 19, 1985

Mr John Stites, page 2

The test method used was as follows:

Photographs were made of each field at 20000 times. The maximum and minimum sizes were measured separately and recorded. Ten additional particles were measured. The mean and standard deviation were determined and recorded and reported in the table above.

Preparation: Each sample was dispersed in N- butanol for one hour in an ultrasonic cleaner. A drop of the dispersion was dried on a microscope stage, coated with gold and photographed in a scanning electron microscope.

Sincerely yours,

Norman M. Hodgkin, Ph.D.